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# STUDY OF THE USE OF PREIMPREGNATED ROVING WITH NUMERICALLY CONTROLLED WINDING EQUIPMENT

ROCKETDYNE

A DIVISION OF NORTH AMERICAN AVIATION, INC. 6633 CANOGA AVENUE, CANOGA PARK, CALIFORNIA

CONTRACT NOW 61-0498-c (FBM)

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BUREAU OF NAVAL WEAPONS SPECIAL PROJECTS OFFICE DEPARTMENT OF THE NAVY WASHINGTON 25, D. C.

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#### ROCKETDYNE

A DIVISION OF NORTH AMERICAN AVIATION, INC.

6633 CANOGA AVENUE CANOGA PARK, CALIFORNIA

Contract NOw 61-0498-c (FEM)

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#### FOREWORD

This final report covers the work performed under Contract NOw 61-0498-(FBM) for the Special Projects
Office, Bureau of Naval Weapons, Department of the Navy.

#### ABSTRACT

The advantages of preimpregnated roving, especially for use in numerically tape-controlled winding equipment, are recognized. This has led to the need for more extensive knowledge of the relationship of materials properties and process parameters as they affect the properties of filament-wound structures. This program of study covered the following items related to use of preimpregnated roving: (1) storage and preheating conditions, (2) winding tension and roving resin content, (3) tackiness and volatile content of the roving strand, (4) preimpregnated roving package design, (5) design of winding equipment components, and (6) voids content in the laminate and variation in strand width. A new tackiness test developed for preimpregnated roving is discussed. A strand tensile strength test on uncured roving was performed and its potential use for quality control purposes discussed. A preliminary process specification for use of preimpregnated roving, employing program results, has been written.



#### ACKNOWLEDGMENT

The work for this program was performed within the Structural Plastics Department and with the assistance of several members of the department. Special acknowledgment is made to John Leigh who assisted in writing much of the material.



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#### INTRODUCTION

Filament winding as a technique for manufacturing high-strength, precision components such as radomes, pressure vessels and rocket motor cases has, in the past, mainly utilized fiberglass roving and liquid resin applied to a mandrel by a mechanical winding machine. The roving has been impregnated with the resin at the winding machine. This system, called the wet winding system, has certain inherent difficulties which can be overcome by the use of roving preimpregnated with resin. Table 1 lists some of the advantages of preimpregnated roving, with the corresponding shortcomings of the wet system. also listed.

The use of mechanically controlled winding machines has a number of drawbacks. In general, these machines utilize a combination of cams, gears, sprocket wheels, or feed screws to control the relative motion between the rotating mandrel and roving delivery device. This requires time-consuming delays to change winding angles or pattern, and involves maintenance of a stock of gears and use of expensive cams, the manufacture of which requires many hours of design calculations and complex machining. Errors in setup or variations in pattern during winding may go undetected until too late to correct the part being wound. These errors could be chargeable to wear on the cams and gears in continuous use. Errors also can occur because of slop and backlash in mechanical linkages. Many mechanically controlled machines are also very limited as to the shape of parts which can be successfully wound.

The use of numerically controlled winding equipment overcomes these disadvantages and, at the same time, offers these many advantages:

#### TABLE 1

## ADVANTAGES OF PREIMPREGNATED ROVING OVER THE WET WINDING SYSTEM

#### Preimpregnated Roving

- 1. Laminates are not subject to large variations in resin content caused by changes in tension.
- 2. Resin content in laminates is affected less by diameter of mandrel.
- All resin systems can be used, even those requiring solvents.
- 4. Long shelf life at room temperature permits fabrication of articles requiring extended processing time.
- Mandrels may be rotated at high speed to allow more rapid fabrication and shorter production time.
- 6. The rowing has good storage life under refrigeration. It requires no special preparation for use.
- 7. Properties of a given lot of roving can readily be determined by means of quality control methods applied before winding operation.
- 8. It eliminates the steps of wet resin impregnation and drainage.
- 9. It is unnecessary to mix resin and hardener with inherent risk of error.
- 10. Preimpregnated roving may be used to wind more unstable patterns without slippage.

#### Wet System

- 1. Laminates are subject to migration of resin to outside layers and subsequently nonuniform resin content throughout thickness of wall.
- 2. Low resin content cannot be obtained on large-diameter parts because of inability to obtain high enough tension.
- Many variables require control to ensure uniform resin content.
- 4. Has limited winding speed because of the risk of throwing resin off the mandrel at high rotation speeds.
- 5. Is inherently messy, requiring time consuming cleanup operations.
- The wet system requires use of weighing and mixing equipment.
- 7. Rapid feed of roving creates impregnation problems.
- 8. The wet system cannot use resin systems that require use of solvent.

- 1. Versatility: A numerically controlled filament winding machine can be programmed for any winding operation within its range by simply changing the control tape. In addition, this allows a series of winding operations to be run in rapid sequence. No time delaying change in mechanical setup is required. Many different shapes can be readily wound.
- 2. Accuracy: Numerically controlled equipment is capable of precise winding patterns. A demonstration of this is shown in Fig.

  1. This pattern was applied by a Rocketdyne numerically controlled machine. Accuracy in pattern laydown is believed to be significant in producing optimum strength properties in filament-wound structures. The ability of numerically controlled equipment to make reproducible filament-wound structures eliminates variables in the performance of these structures.
- operation Speed: The use of digital programming elements and rapid hydraulic drive mechanisms on the Rocketdyne filament winding equipment will provide high operational speeds to meet any production requirements.
- 4. Quality Control. The accuracy and reproducibility of filament—wound patterns fabricated on numerically controlled equipment contribute to the effectiveness of quality control procedures.

  The recording equipment associated with these machines will also assist in quality control procedures.
- 5. Storage: Any winding pattern can be readily stored in the form of a deck of punch cards and/or digital tape for any required future use. This, in effect, means that numerically controlled winding equipment can be immediately staged for any operation for which a control tape has been previously prepared.



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Test Pattern Applied Using Numerically Controlled Winding Machine Figure

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Because of the advantages listed, it was decided that filament winding of efficient structures could best be accomplished by using preimpregnated rowing with numerically controlled equipment. It was also realized that attainment of these efficient filament-wound laminates using preimpregnated rowing and numerically controlled equipment would require factual information concerning the materials and processes involved. This need has led to the following program.

#### **OBJECTIVES**

The program undertaken has as its objective the development of optimum processing methods for fabricating rocket motor cases, tanks, and other parts from preimpregnated roving using numerically controlled equipment.

This objective was accomplished by performance of these activities:

- 1. Determination of the properties required of preimpregnated roving for its most efficient use with numerically controlled winding machines
- 2. Determination of the most favorable conditions for handling preimpregnated roving both in storage and during fabrication
- 3. Determination of winding machinery modifications required when preimpregnated roving is used in conjunction with high-speed numerically controlled winding equipment
- 4. Writing standard procedures for quality control and fabrication methods for the use of preimpregnated roving with numerically controlled machinery, and incorporating these procedures into a process specification

Items 1 through 3 were approached through a testing and evaluation program using cylindrical filament-wound test cylinders and wound vessels per ABL print 6400A as the test items. Data obtained were used in writing a process specification which describes requirements of preimpregnated roving, machinery, and processing methods for the use of preimpregnated roving with numerically controlled equipment.

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The testing and evaluation program encompassed these areas of investigation:

- 1. Resin advancement as it occurs during various storage conditions and preheating conditions
- 2. Winding tension and resin content as they affect properties of laminates
- 3. Tackiness and volatile content as they affect processing conditions
- 4. Packaging and how it affects processing
- 5. The effect of voids content and variation in strand width on the strength of laminates
- 6. Optimum design of winding equipment components

Results obtained in each area will be covered in the following discussion.

The parameters of principal interest were those which can be controlled during the winding process. It is recognized that many of these parameters are interrelated. However, the test procedures were, in most cases, limited to evaluation of simple interactions such as the effect of winding tension on resin migration. Multiple factor correlation of four or five variables on a simultaneous basis was not attempted in this program. Rocketdyne's numerically controlled filament winding machines were specifically designed to use preimpregnated roving and were used for the winding of ABL qualification tanks and the study of optimum winding methods. Various types of preimpregnated roving handling equipment were studied for use with this machine.

Study of resin systems or fiberglass materials was not an objective of this program. Consequently, the entire test and evaluation program was performed with one material system.

#### SUMMARY AND CONCLUSIONS

In this program, Rocketdyne has conducted an evaluation of some of the basic properties of preimpregnated roving and its use in numerically tape-controlled winding equipment. The emphasis in this study was to determine the practical considerations related to the use of these materials in production situations.

#### EVALUATION METHODS

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Investigation of the various materials properties and process parameters was accomplished in most instances by fabrication of two test sample configurations, as follows:

- 1. A cylindrical sample 3 inches in diameter by 6.37 inches long. This specimen was tested by hydrostatic pressure to burst failure. Ultimate hoop tensile stress was calculated, using the pressure at failure. Another test applied to the cylinder was parallel interlaminar shear performed on small ring segments cut from the sample.
- 2. A pressure vessel 18 inches in diameter by 24 inches long. This case conforms to the ABL6400A design. Testing was by hydrostatic pressure test to burst failure.

Two other tests used were developed during the investigation. These are a strand tensile strength test and a test for tackiness of the preimpregnated roving strand. In the latter, a number value indicating the tackiness of the roving results from performance of the test.

#### MATERIALS

Because of limitations on available funds, only one resin system could be used throughout the program. The material chosen for the specimen studies was ECG140-20 end roving with Owens Corning 801 sizing impregnated with E787, a proprietary epoxy resin system of U. S. Polymeric Chemicals, Inc. E-HTS fiberglass roving impregnated with the same resin was used for the studies involving the 18-inch-diameter cases.

#### STORAGE AND PREHEATING CONDITIONS

An extensive program was conducted to determine the effect of various conditions of storing preimpregnated roving preliminary to the winding process. Evaluations were made of the continued usefulness of roving stored at (1) room temperatures at 70 to 90 F, (2) refrigerator temperatures 40 to 50 F, and (3) deep-freeze temperatures -10 to ÷10 F.

Evaluations of the rowing were made on 3-inch-diameter x 6.37-inch-long cylinders tested to burst by hydrostatic pressure. The test cylinders were fabricated under three conditions during the winding operations:

- 1. The cylinders were wound on a heated mandrel, followed by an oven cure. Mandrels were preheated to 150 F.
- 2. The roving strand was heated during the winding operation with the mandrel at room temperature, followed by an oven cure. Temperature of the roving was approximately 125 F.

3. The roving strand was wrapped cold (without any heating of the strand or mandrel), followed by an oven cure. The following conclusions were reached from this 6-month evaluation.

Storage at room temperatures (75 to 85 F) resulted in deterioration of the material within 7 days. (For the purpose of evaluation, preimpregnated roving was considered to have deteriorated or be nonusable when it gave hoop tensile strength values with the 3-inch-diameter cylindrical specimens below the average range of values obtained from fresh material.)

Storage at refrigerator temperatures (35 to 45 F) was effective for periods up to 6 months. The preimpregnated roving gave hoop tensile strength values equivalent to the average range of results obtained from fresh material throughout the evaluation. However, some loss in strand pliability and tackiness was noted after approximately 3 months.

Preimpregnated roving can be stored at deep freeze temperatures (-10 to : 10 F) for periods up to 6 months and still give hoop tensile strength values equivalent to fresh material. In addition, there was no apparent loss in strand pliability or tackiness after the materials were brought to run temperature.

Preheating of the mandrel or material will extend the useful life of material stored at room temperature up to 26 days (conclusion of test). The heat restores the tackiness and pliability of the roving and provides for proper consolidation during winding.

Preheating the mandrels or material when using "fresh" material does not improve the physical strength of the laminate over that obtained without preheating.

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It is recommended, when using the E787 resin system in a preimpregnated roving, that the material be stored at deep-freeze temperatures to ensure that the material remains fresh, and that no preheating need be used with this material.

#### WINDING TENSION AND RESIN CONTENT

The objective of this program was to determine the effects of strand winding tension, resin content, and resin migration on the properties of filament-wound laminates. Evaluation was performed by winding 3-inch-diameter cylinders and testing for hoop tensile strength and interlaminar shear strength. In the resin migration studies, 2-inch-wide bands were wound on various-diameter mandrels.

The effects of roving resin content and winding tension on hoop tensile strength and interlaminar shear strength can be summarized as follows:

- 1. Optimum tensile strength is obtained with roving which has a resin content of 17 to 20 percent and has been wound at 0.8 to 1.0 pound per end on a 20-end strand.
- 2. Optimum shear strength is obtained when winding with a strand tension of 0.8 to 1.0 lb. per end on a 20-end strand, the same as for tensile strength.

However, maximum shear strength was obtained with roving having a resin content of 26 percent. Since the optimum values of tensile strength and interlaminar shear strength are obtained with roving at different resin content, the choice of resin content must depend upon which property is most critical in the application.

1.2



Studies were made of the combined effects of tension, resin content in the roving, and mandrel diameter as they affect resin migration. Resin migration was defined as the excess resin that is forced to the surface of the part during fabrication and heat cure. It was noted that resin migration increased rapidly with tension on a 3-inch-diameter mandrel, and was much less on larger-diameter mandrels up to 13 inches. No relationship with resin content was discernible. The factors of winding tension (T) and mandrel diameter (D) were combined into a radial load factor (P =  $\frac{T}{D}$ ) and plotted against resin migration to clearly show the trends described. It was also noted that the outer layers of a laminate acquire a higher resin content than the inner layers because of migration of the resin. However, no relationship between resin migration and tensile strength was discernible.

#### TACKINESS AND VOLATILE CONTENT

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The objective of this study was to measure the effects of strand tackiness and volatile content upon the processing of preimpregnated roving and the mechanical properties of cured laminates.

A test apparatus was developed for measuring the degree of tackiness in uncured roving. This device uses an inclined V-shaped ramp and V-shaped horizontal tracks. A steel ball is allowed to roll down the ramp onto the track, on which are stretched two parallel strands of roving. The ball rolls along the horizontal section in contact with the strands of roving. The tackiness of the roving is measured by the distance the ball rolls. The nature of the test is such that a short distance indicates more tack than a long distance. Tackiness is reported as a number which is the distance that the ball rolls along the track.

Apparently inconsistent results were obtained in the first experiments with the tackiness tester since it proved difficult to evaluate relatively dry or nontacky preimpregnated roving. However, later work in this area showed that this method gives reproducible results in measuring tackiness in fresh or tacky roving. Using the rolling-ball tackiness tester, the level of tack may be expressed by a quantitative number.

A series of experiments to measure the effect of volatile content on tackiness was made on preimpregnated roving stored at room temperature. Although the volatile content of the samples decreased with time of storage, no correlation could be found among the factors of volatile content, resin content, and tackiness measurements. One possible explanation of these results is that polymerization of the resin with aging at room temperature may be the controlling factor.

Tackiness in the rowing was found to be an important factor in winding an unstable pattern. A series of experiments was conducted to determine this relationship. Rowing having two degrees of tackiness, as measured by the backiness tester, was used. A series of windings was placed on an 18-inch-diameter by 24-inch-long mandrel. Each set of windings was placed at a more unstable pattern position until slip occurred. Greater resistance to slip by more tacky rowing was effectively demonstrated. A method of predicting slippage when winding an unstable polar-type pattern was developed. Evidence was gathered of the need for using very pliable and tacky preimpregnated rowing when winding a very low-angle helical pattern using the numerically tape-controlled machine to prevent slippage and to obtain a high degree of accuracy.

#### **PACKAGING**

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The objectives of this study were (1) to determine the optimum method of packaging preimpregnated roving, and (2) to determine the best method of applying tension to the strand during winding operations in relation to the type package used.

A 75-degree way wind (helical wrap) on the package and a straight wind (circumferential wrap) on the package were two package designs studied.

Two methods of applying tension during winding operations were used, one in which brakes were applied to the package spool, and one in which tension was applied to the strand by a set of brakes after it left the spool. In the latter, the package spool was allowed to turn freely.

Evaluation of the packages and methods of applying tension was performed by measuring degradation (if any) on the roving strand. Degradation of the shrand under the various conditions was determined by winding 3-inch-diameter x 6.37-inch-long cylinders which were tested to burst by hydrostatic pressure.

Applying tension by brakes on the spool produced no visible effect on the 75-degree way wind package. However, with a straight wind (circumferential wrap) package consisting of fresh, pliant roving, the soft strand dug into the package surface between adjacent strands. Aged or dry roving did not act in this manner, but unwound from the spool without digging into the package.



With a 75-degree way-wind wrap, test cylinders made with tension applied to the strand had slightly higher hoop tensile strengths than the equivalent cylinders made with braking action on the package spool. This indicates that there was some strand degradation when brakes were applied at the spool. Equivalent tests were not completed on a parallel-wind package, to compare the effects of applying tension at the spool and on the strand, because of lack of material. The results of this study indicate that the application of tension to the strand is preferred to applying tension at the spool.

Several advantages and disadvantages of each package design were noted. However, no firm recommendation as to the desirability of one over the other can be made. More extensive use of each in volume production operations is needed before making a reliable decision. It is likely, however, that a package with a greater wayward angle, but not quite parallel, would be the best compromise.

#### WINDING MACHINE COMPONENTS

This area of the program had the following objectives:

- Studying accessory equipment for handling preimpregnated roving on NTC winding machines (The accessory equipment to be studied included (1) plastic materials for pulley and guide surfaces,
   (2) preheating equipment, and (3) tensioning and slack-takeup devices using mechanical, magnetic, and hysteresis brakes.)
- 2. Determining the optimum method of applying tension to a strand during winding operations
- 3. Developing methods for maintaining controlled strand tensions on preimpregnated roving during high-speed winding operations

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- 4. Developing optimum methods and equipment for handling multiple strands of preimpregnated roving in winding operations
- 5. Determining the accuracy of pattern laydown using numerically controlled winding equipment

Investigations made in support of the objectives are as follows.

#### Multiple Pulley Systems

The effects of multiple pulley systems on preimpregnated roving strand were evaluated in a series of tests. Comparative evaluations were made from 3-inch-diameter test cylinders, tested to burst under hydrostatic pressure. Any damage to the winding strand in passing over the various pulley systems could be shown by reduction in hoop tensile strength from standard values.

Results of the tests on multiple pulley systems indicate that preimpregnated rowing may be degraded when passing through a large number (10) of pulleys. Consequently, use of as few pulleys in the delivery system as possible is recommended.

#### Ceramic Guide Bushings

The use of ceramic eye guides or bushings was also evaluated by tests on 3-inch-diameter cylinders. Preliminary results from these tests showed no apparent degradation of the strand when passed under 20 pounds of tension straight through a ceramic eye. However, when the strand was bent at a

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sharp angle around the edge of a ceramic eye during the winding operation, excessive strand friction was noted and caused filament abrasion as well as erratic variations in winding tension. The use of ceramic guide bushings to direct strands of preimpregnated roving under tension is not recommended.

#### Materials for Pulleys and Guide Devices

Sinel pulleys and guide rollers showed no tendency to be abraded by the glass strand. However, metal surfaces showed a serious tendency to pick up resin, with eventual shredding and degradation of the preimpregnated roving strand.

Plastic materials were the most satisfactory for guide pulleys. Nylon had the best combination of wear resistance and low tendency to pick up resin. Teflon and Kel-F were excellent in this respect but had poor resistance to wear which required frequent expensive replacement.

#### Prehearing Equipment

Preheating equipment was produced for use with the aging study. This equipment heated the roving, just prior to placement on the mandrel, by passing it through a chamber heated by a hot air flow. The equipment was satisfactory, but the study indicated that preheating of the roving is not normally required.

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#### Tensioning Devices

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Simple mechanical brakes were adequate where strand feed velocity was nearly constant. An air-controlled clutch which allows dynamic friction loading to be relatively stable over a wide range of strand feed velocity was found insufficiently sensitive for low-end-count strands where the total tension load was low. At high-tension loads the clutch was satisfactory. Study of a magnetic clutch indicated similar results. However, the tension of the strand as delivered to the mandrel is also dependent upon the entire delivery system. Complete definition of system requirements was found to be beyond the limits of this program.

#### OPTIMUM TENSIONING TECHNIQUE

It was determined that the tension should be applied to the strand rather than the roving package, regardless of the design of the package.

#### CONTROLLED STRAND TENSION

This study is related to the tensioning devices and delivery system used; considerably more effort beyond the scope of this program is required to specify adequate methods for obtaining controlled strand tension.

#### METHODS FOR WINDING WITH MULTIPLE STRANDS

A wide band consisting of two strands of roving was used without difficulty to fabricate an 18-inch-diameter case. Hydrostatic burst test performance of the case was comparable to one of similiar construction made with a single strand of roving. The use of multiple strands made into a single, wide band is readily conceivable.

#### ACCURATE PLACEMENT OF STRANDS DURING WINDING OPERATIONS

Test patterns have been wound with thin white thread on the numerically tape-controlled winding machine developed, under company funding, by Rocketdyne. Measurements of the accuracy of strand placement showed spacing variations of the order of 0.01 to 0.02 inch. This degree of precision indicates that the method of machine control is a practical approach.

#### QUALITY CONTROL

The objective of this part of the program was to determine suitable methods for maintaining quality control standards on preimpregnated roving used in production applications.

A method for testing strand tackiness was developed and can be applied as a quality control procedure.

A strand tensile test procedure was developed for determining the strength and/or presence of catenary in preimpregnated roving material received. A series of experiments were performed, using this procedure, with these results:

- 1. Test values obtained on material from the outside of the spool are probably indicative of the quality throughout the spool.
- 2. There is no correlation between strand strength and resin content.
- 3. It is possible to detect catenary in the fiberglass roving with this test.

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Additional work should be performed to relate strand tensile strength to strength in a filament-wound case.

Data from the extensive number of hoop tensile strength tests performed during the program can be used as the basis for quality control standards.

#### CASE WINDING STUDIES

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In this phase of the program, studies were made of the following:

- 1. The effect of voids and gaps on the strength of laminates
- 2. The effect of roving band width on the stress level in a laminate
- 3. Comparison of the use of the NTC machine with mechanically controlled equipment

These studies were performed by fabricating 18-inch-diameter by 24-inch-long cases. All were wound on the numerically tape-controlled equipment and, subsequently, tested hydrostatically.

#### Voids and Gaps

Two cases of basically similar construction were made; one case was made with gaps between the roving strands to create voids in the case, and one was made without gaps. After cure of the cases, no voids were visible because of resin flow that filled the gaps. Hydrostatic test performance of the cases was nearly identical, indicating, at least on the cases tested, that gaps which fill with resin have no effect on strength. Further tests would be required to confirm this and to investigate the effects of voids.

#### Roving Band Width

Two cases were made with a wide roving band (0.125 inches compared to a normal width band of 0.085 inches). The hydrostatic test performance of each was compared to cases of basically similar construction which were made with the narrow-band roving. A significant improvement was noted where the wide band was used--all of which could not be attributed to use of the material only. The particular preimpregnated roving used has considerable variation in bandwidth. Some benefit conceivably could be derived from the use of wide-band roving, although more tests would be necessary to confirm this.

#### NTC vs Mechanically Controlled Equipment

Two cases made on the NTC machine were compared to the ABL 10 case, made on the mechanically controlled machine during another program. All were of basically the same construction and made from the same material, except wide-band roving was used on one of the two cases. The ABL 10 case was considered the best of its kind prior to this program. Both cases developed higher glass stress in the longitudinal filaments where all failed during hydrostatic testing. The remaining cases made on the NTC machine as part of this program were of a slightly different construction. However, they all developed higher glass stress for a similar type test failure than did the ABL 10. One of the two test cases referred to originally, i.e., case S/N 5, developed a glass stress of 435,000 psi in the hoop fibers without the occurrence of failure in this area. This compares to an ultimate of 500,000 psi for the E-glass monofilament. Use of the NTC machine in this program has definitely produced superior filament-wound structures.

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#### DISCUSSION

#### **EVALUATION METHODS**

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Various processing parameters and materials properties were investigated. This was accomplished, in most cases, by the fabrication of cylindrical test specimens. This specimen is 3 inches in diameter by 6.37 inches long, and has been a standard test configuration at Rocketdyne for several years. The winding pattern used to fabricate the cylinder was a straight circumferential wrap. The results of the various investigations were evaluated by performing certain tests on the cylinder. The one most commonly used was a hydrostatic pressure test from which the ultimate hoop tensile stress was calculated. Details of the test specimen and test procedure are given in the Appendix.

Another test used to evaluate results of the investigations was interlaminar shear. Again, the cylindrical sample was used. Construction of the cylinder was similar to that used for the hydrostatic test sample. Small ring segments were cut from the cylinder and tested in parallel shear. Details of this test procedure are given in the Appendix.

Two other tests were used during this investigation. One was a strand tensile strength test. In this test, a short strand of preimpregnated rowing is submitted to a tensile load until failure. This test was developed primarily as a means of quality control and is discussed in more detail under that section. The other test was one developed for measuring the

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level of tackiness in the roving strand. A number value indicating the tackiness of the roving results from performance of the test. Details of the tackiness test are presented in the Discussion section.

In addition to the cylindrical test specimen mentioned, an 18-inch-diameter by 24-inch-long pressure vessel was used in some of the following investigations. Details of design (Fig. 2) of this vessel are defined by Allegheny Ballistics Laboratory drawing ABL 6400A. Results of applying various parameters to the fabrication of the ABL cases were evaluated by hydrostatic testing. Wall stresses were determined, following test, and evaluated in relation to the variables applied.

#### MATERIALS

Investigating resin systems or fiberglass materials was not a program objective, so no materials investigation program was performed.

Previous testing and evaluation by Rocketdyne led to the choice of ECG 140-20 end rowing with OC801 size, and an epoxy resin, as the most suitable material for use in this program. The resin system used was E787, a proprietary system of the material producer.

The preimpregnated rowing material selected for evaluation in the program is produced by U.S. Polymeric Chemicals Inc., Santa Ana, California. The choice of this material was based on the following considerations:

1. Epoxy-impregnated-type material has the widest current applicability for use in rocket casings and propellant tanks.

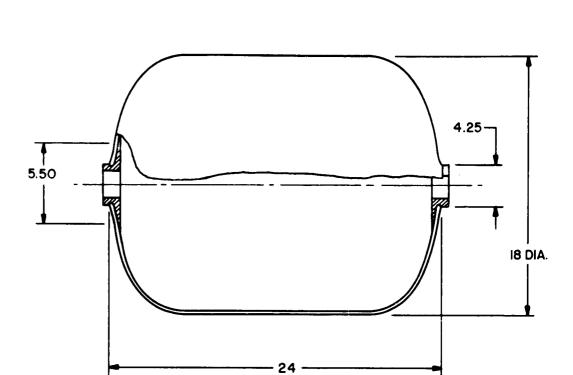


Figure 2 . Test Case, ASL 6400A

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- 2. This particular roving material showed consistently superior properties when previously fabricated by Rocketdyne into tanks and test cylinders.
- 3. The preimpregnated roving manufactured by U. S. Polymeric Chemicals is considered to be typical of material made in large production runs. (The intent of this project is to test the type of material that would be manufactured in large lots for use in heavy production operations.)

This material was used for all phases of the test and evaluation program except the fabrication of the 18-inch-diameter cases. For these items, E-HTS fiberglass roving impregnated with the same resin system was used. The E-HTS glass permitted making comparisons of cases fabricated in this program with similar cases previously fabricated by Rocketdyne as part of a qualification program for ABL. The referenced cases were made using the E-HTS glass roving.

#### TESTING AND EVALUATION

The objectives of this program were accomplished by the study of certain process parameters and material characteristics. A discussion of these areas of investigation and the results obtained follows.

#### Storage and Preheating Conditions

This part of the program consists of an aging study. Portions of a single batch of preimpregnated rowing were stored at three different conditions:

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(1) room temperature (75 to 85 F), (2) refrigerated temperature (35 to 45 F), and (3) deep-freeze temperature (-10 to +10 F). Samples were removed from storage at intervals to determine the shelf life limitations of the material at each condition of storage

For each storage condition, standard 3-inch-diameter cylindrical samples were wound, using three processing conditions: (1) where the mandrel was heated to 150 F in an oven just before winding (2) where the roving was preheated to 125 F and wound on a cold mandrel, and (3) where roving at room temperature was wound on a mandrel at room temperature. Roving was heated by passing through a metal pipe into which heated air was introduced. Figure 3 shows this apparatus and the winding machine and a mandrel at the beginning of a wrapping operation. Evaluation of the parameters involved has been performed by winding 3-inch inside diameter sample cylinders which are subsequently burst nested to determine the hoop tensile strength. Three cylinders were fabricated for each specific aging and preheating condition investigated. Each point on the graphs is the average value of the test values obtained on the three samples fabricated for that particular set of conditions. Variations in the applied parameters have resulted in variations of physical strength in the test specimens, indicating the effect of the parameter upon the winding operation.

Hydrostatic burst tests were performed on cylindrical samples made with the roving stored at room temperature for various lengths of time up to 25 days. Results of these tests are plotted in Fig. 4. The useful life of material wound at room temperature (condition 3) is approximately 7 days. This can be extended to at least 3 1/2 weeks (testing arbitrarily stopped here) by heating the roving as it is wound on the mandrel (condition 2).

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Figure 3. Cylindrical Specimen Winding Machine With Roving Heating Device

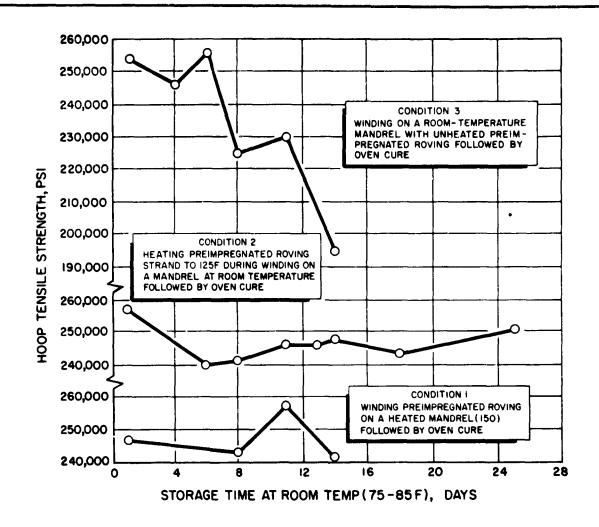


Figure 4. Effect of Room Temperature Storage of Preimpregnated Roving and Process Parameters of Hoop Tensile Strength

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Useful life of the preimpregnated roving can also be extended by heating the mandrel (condition 1). The effective limits of this method could be determined by additional testing. It should be noted that the room temperature aging was begun one week after the preimpregnated roving was manufactured. In the interim, the material was stored in the deep freeze where it was assumed that no adverse deterioration of the material occurred.

Hydrostatic burst tests were performed on cylindrical samples made with the roving stored at refrigerated and deep freeze temperatures. Graphs (Fig. 5 and 6) of the results of these tests indicate that the material has a useful life of up to 6 months when stored at the above temperatures. However, material stored in the refrigerator tends to lose some of the original tackiness in approximately 8 to 12 weeks. This does not occur with material stored at deep-freeze temperatures. Consequently, deep-freeze storage is preferred. The graphs also indicate that preheating the roving or the mandrel does not improve the strength properties obtainable with fresh roving that has adequate flow, pliability, and tack. There is some indication that preheating does tend to reduce wide fluctuations obtained on unheated material.

During the period when winding of the specimen using preimpregnated roving stored at room temperature was being performed, several observations were made. It was noted that as the roving aged it became less tacky and, eventually, not only dry to the touch but was also advanced in pre-cure so that the strand would crack and the resin would powder when bending the strand. This occurred in approximately 8 to 10 days. As the roving aged, wrinkles caused by the turnaround point on the package became permanently

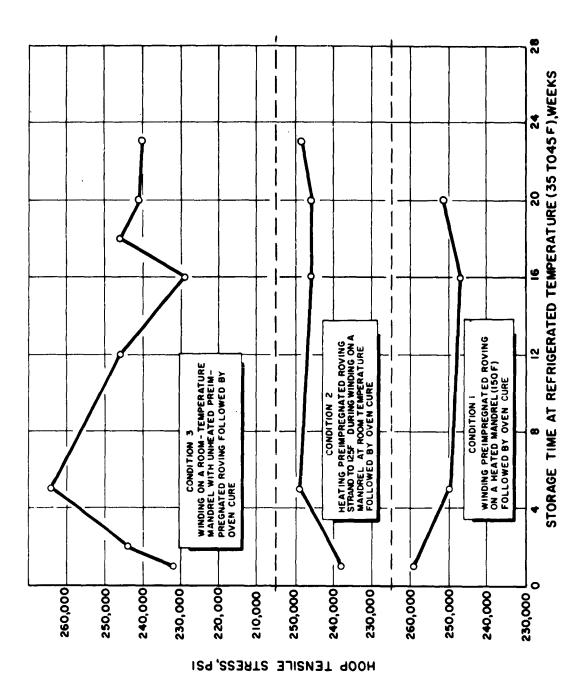
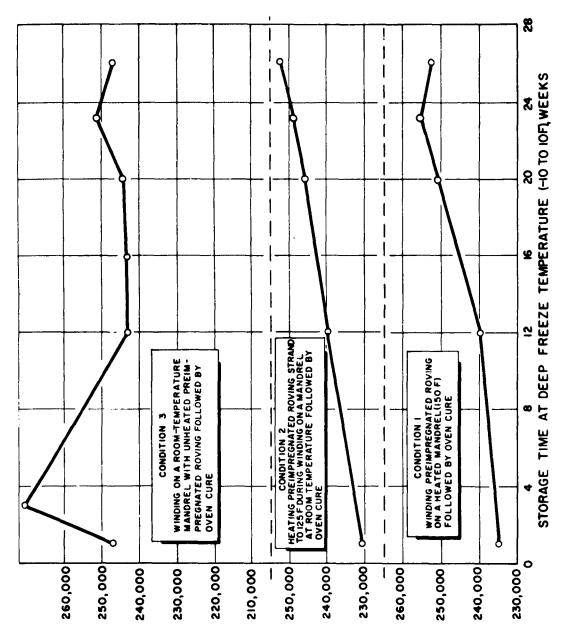


Figure 5. Effect of Refrigerated Storage of Preimpregnated Roving and Process Parameters on Hoop Tensile Strength

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HOOP TENSILE STRESS, PSI

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Effects of Deep-Freeze Storage of Preimpregnated Roving and Process Parameters on Hoop Tensile Strength

Figure 6.

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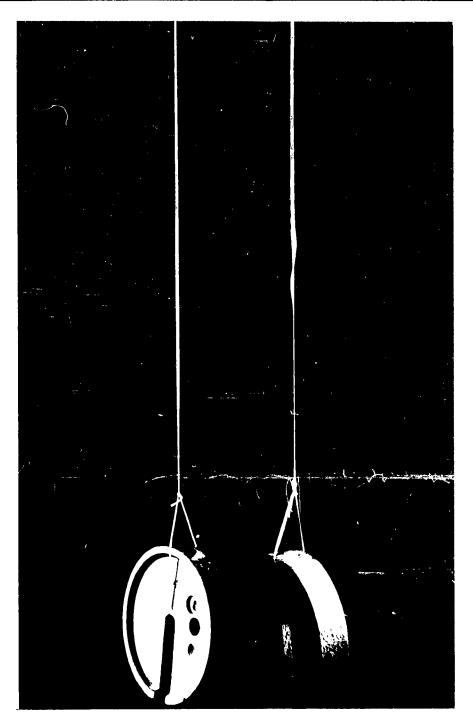
set, even under tension. An example is shown in Fig. 7 which contrasts a fresh roving strand to an aged roving strand. Wrinkles of this nature disappear upon heating the strand.

Cylindrical samples wound with roving aged at room temperature for 14 days have a dry, ropey appearance when they are wound at room temperature on a mandrel at room temperature. When wound on a heated mandrel, or when preheating the roving, the resin softens and consolidation of the roving occurs; improvement in appearance is concomitant.

This difference in appearance before curing is shown in Fig. 8 where the sample wound on a heated mandrel is on the left, the heated roving sample is in the center, and the sample wound with roving and mandrel at room temperature is on the right. The same samples after cure are shown in Fig. 9. The difference in appearance is still apparent, but to a lesser degree (Samples are shown in the same order). Another aspect of the aging phenomenon is the variation that exists in the roving from the outside to the inside of a spool. Figure 10 shows three samples made from a spool aged 14 days at room temperature. The sample on the left was wound using material from the outside of the spool, the sample in the center of the photograph was taken from the center, and the right-hand sample was taken from the inside of the spool. The material from the inside of the spool was more tacky and pliable, and the resin flowed more during oven cure.

From the above, it may be concluded that preimpregnated roving is best shipped and stored under deep-freeze conditions. It is also apparent that these materials can be readily stored to meet production needs for periods up to 6 months when stored at deep-freeze temperatures.

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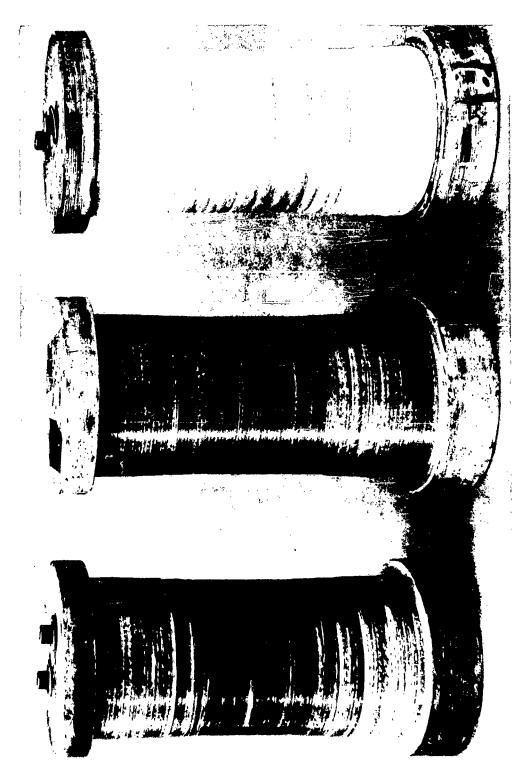


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Figure 7. Strands of Preimpregnated Roving Showing the Permanent Twist and Wrinkles Created in the Package of Aged Material and Not Present in Fresh Material

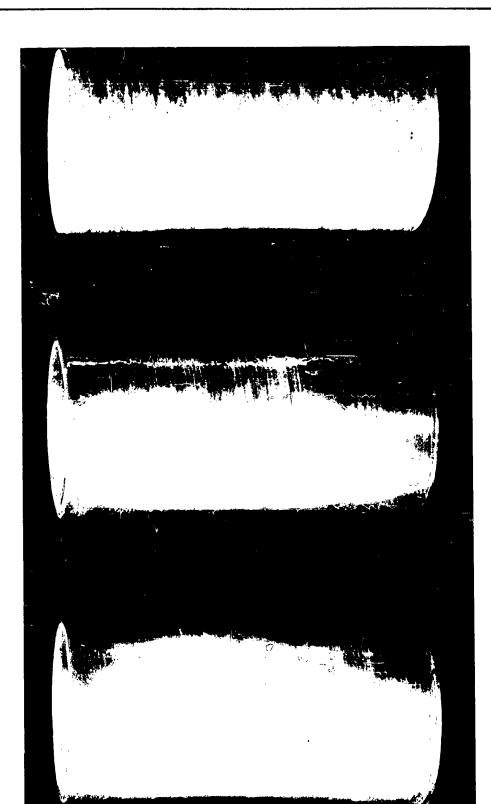
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Uncured Specimens Wound With Aged Preimpregnated Roving Using Three Processing Techniques



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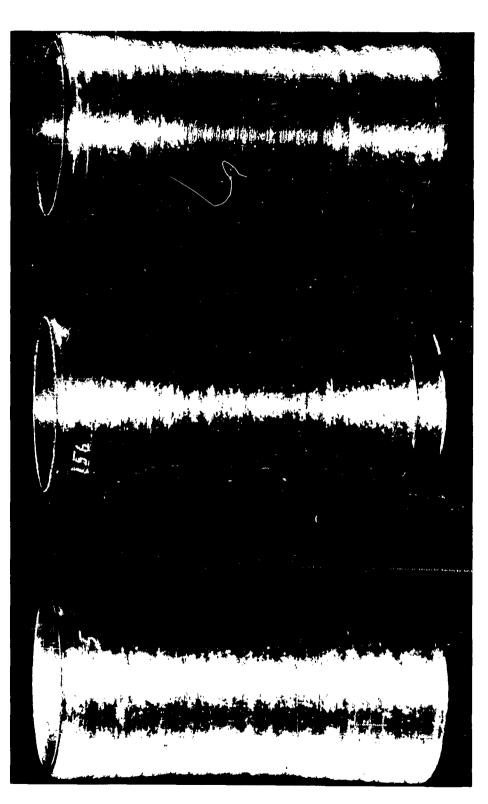
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Figure 9. Cured Specimens Wound With Aged Preimpregnated Roving Using Three Processing Techniques

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Heating the mandrel or rowing strand during winding operations did not improve the hoop tensile strength properties of test cylinders made from fresh rowing which has adequate tack and flow. Preheating the rowing strand or the mandrel did improve the strength properties of test cylinders made from over-age resin.

Preheating the strand or mandrel caused an increase in strand pliability and increased tackiness. It also appeared to assist in the consolidation of the resin and therefore can be used to extend the useful life of pre-impregnated roving. These effects may be desirable enough, in some applications, to justify the use of preheating methods. For example, an increase in tackiness may be effective in preventing strand slippage on a mandrel in nongeodesic winding patterns. However, preheating is not recommended (based on the data accumulated) for normal operations. The particular pre-impregnated roving used here consequently should be stored at deep-freeze temperatures to ensure maintenance and use of material in a fresh condition.



#### WINDING TENSION AND RESIN CONTENT

The objectives of this program were to determine the effects of strand winding tension, resin content and resin migration on the properties of parts fabricated from preimpregnated roving.

This area of investigation proved to be or great interest since important interactions were determined for the relationships among resin contents, part diameters, laminate thicknesses and winding tensions. In this study, evaluation was made by determining the effects of the variables on hoop tensile stress and interlaminar shear stress.

Samples were fabricated using preimpregnated roving at four different resin contents and four variations in winding tension. Three cylindrical samples were fabricated for each specific set of conditions investigated. Each point on the graphs summarizing the results represents the average value of three samples for the tensile strength tests and five values for the shear tests. All samples were fabricated and tested at room temperature.

The effects of resin content and winding tension on hoop tensile strength are shown in Fig. 11 and 12. These results indicate that optimum tensile strength is obtained with preimpregnated roving which has a resin content of 17 to 20 percent and which has been wound at 0.8 to 1 pound per end on a 20-end strand.

The most desirable resin content in the roving is indicated to be 17 to 20 percent. Tensile strength drops off greatly below 17 percent except when high winding tension is used. This is probably caused by poor consolidation of the roving because of inadequate radial pressure on the layers of roving. Since radial pressure decreases with increasing diameter, this condition of

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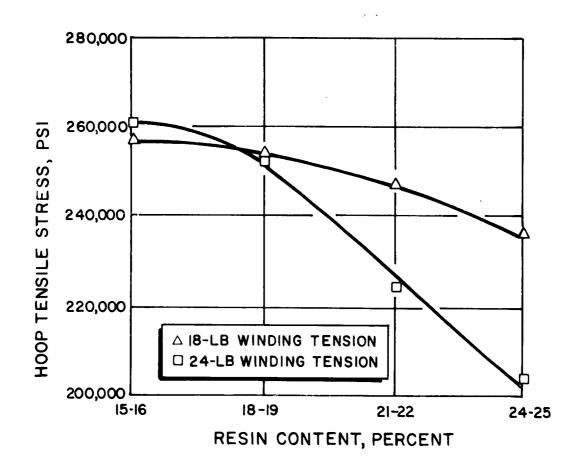


Figure 11. Effect of Roving Resin Content on Hoop Stress of 3-inch-Diameter Cylinders

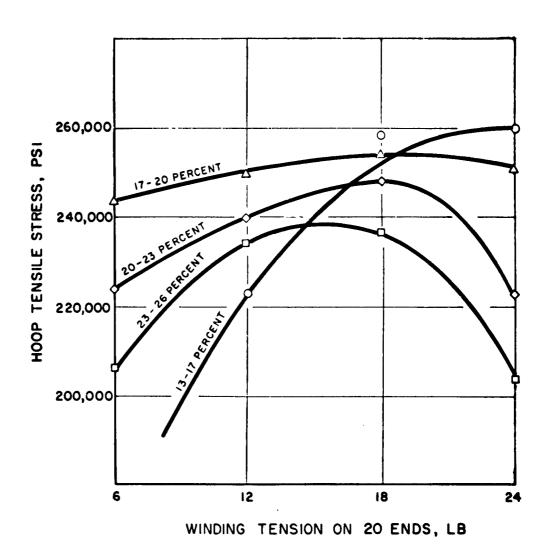


Figure 12. The Effect of Winding Tension and Roving Resin Content on Hoop Stress of 3-Inch-Diameter Cylinders

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poor consolidation would get worse with increasing diameter of part. The tensile strength of cylinders made with roving which has 17 to 20 percent resin content is less sensitive to winding tension, and, therefore, to diameter of part. Consequently, the 17 to 20 percent resin content range is preferred when maximum tensile strength is desired. With the higher resin content rovings, more resin is put into the laminate; the weight then is increased without adding structural support, and the calculated wall stress is effectively reduced.

The effects of tension and resin content on interlaminar shear stress were somewhat different. The results of this investigation are shown graphically in Fig. 13. Interlaminar shear strength at 18 pounds winding tension increased sharply with increased resin content in the roving in the range from 16 to 28 percent. However, the high resin content at which maximum shear value was obtained is not desirable for maximum tensile stress, and the designer using these data must make his choice of materials based on the most critical requirement. Fortunately, the best property values were obtained at the same value of winding tension, i.e., 18 pounds on the 20-end strand. The effect of tension is again to force out trapped air for improved consolidation. This results in a resin structure that is more continuous (fewer discontinuities caused by trapped air) and capable of transmitting shear loads more effectively. Increasing the resin content from an unsatisfactory low value to a higher value helps in obtaining a more continuous resin phase and provides for the improved interlaminar shear values reported.

An example of the effect of resin content on shear strength was noted during hydrostatic testing of the samples for determination of hoop tensile stress. It was observed during testing that most of the cylinders failed as the right-hand view of Fig. 14 shows. However, many of the cylinders made with low-resin-content roving failed as the left-hand view shows. The different

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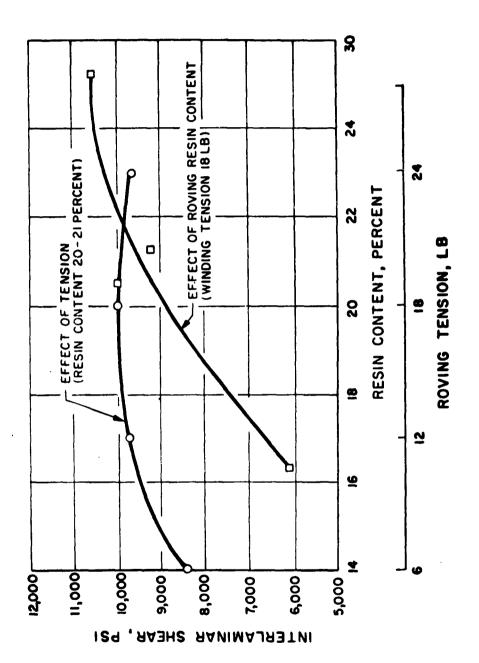


Figure 13. Effect of Tension and Roving Resin Content on Interlaminar Shear

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Figure 14. Mode of Failure of Hydrostatically Tested Specimens

mode of failure seems to reflect the differences in shear strength. During pressurization the cylinder expands radially, more so in the central portion than at the ends where the extra thickness of windings has been placed. This introduces longitudinal bending loads and shear loads. Apparently, failure occurs by the resin failing in shear (the form of failure would be a slit between adjacent windings); the photograph shows the failure was not one of tension in the fibers. It appears from this experience that premature failure can result from low shear strength caused by too low a resin content.

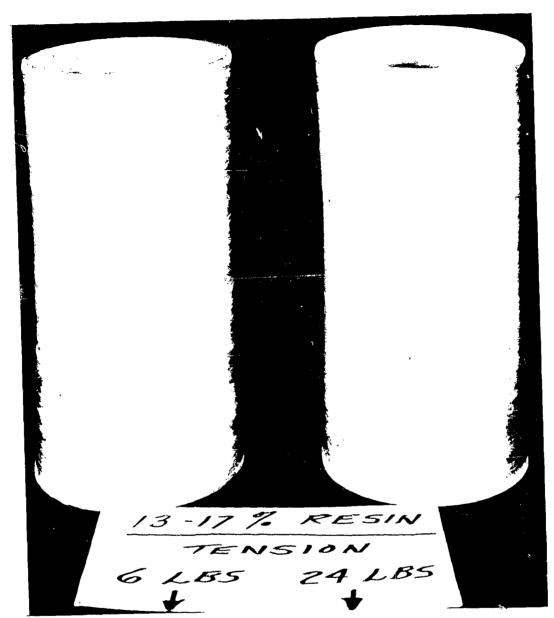
During fabrication of the test cylinders for this study, it was noted that resin migration to the surface was influenced by the resin content in the roving and the amount of tension applied to the strand. The observed condition was recorded in Fig. 15 and 16. Resin migration was more evident with increased winding tension. The effect of tension was also visually more evident for samples with a low resin content than for those with high resin content.

The preceding data and discussion on shear strength apply to 3-inch-diameter cylinders. In the resin migration studies discussed below it was determined that part diameter and thickness affect the properties of a laminate. This is probably true of the shear strength and tensile strength, as well. It is necessary, then, to extend this investigation to larger diameter parts. This extension was beyond the scope of the present program, but is necessary to permit preparation of a complete set of process parameters for a specification.

The experiments above have demonstrated the influence of winding tension on the strength of cylindrical samples. It was noted at the same time that tension influenced resin magnation. Consequently, it was thought that a relationship might exist between resin migration and the strength of a filament-wound structure. A program was subsequently initiated to investigate this relationship.

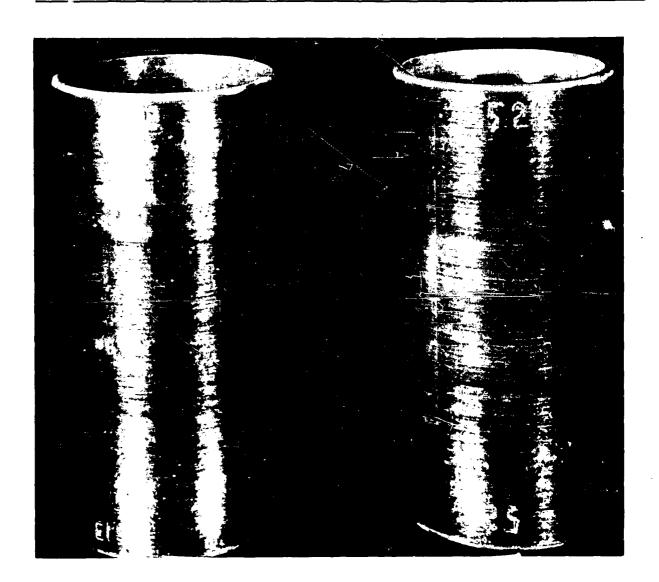
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Figure 15. Cylindrical Samples Showing the Effect of Winding Tension on Resin Migration (13 to 17 percent resin)



6-Pound Tension

24-Pound Tension

16 to 20 Percent Resin

Figure . Cylindrical Samples Showing the Effect of Winding Tension on Resin Migration (16 to 20 percent resin)

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### Resin Migration

Studies were made of the combined effects of roving resin content and tension as they affect resin migration. During the winding operation, tension is applied to the roving strand. This results in a radial pressure similar to that applied by a vacuum bag or platens on a press when laminating fiberglass fabric. The applied pressure in all of these instances results in resin flow when heat is applied during cure. In the filament-wound structure the resin flows to the surface of the structure. This occurs when using preimpregnated roving, as well as with the wet winding system, although to a lesser degree. The surface resin cures in place and forms a relatively smooth, glossy crust. This surface crust is called the migrated resin.

Determination of the amount of migrated resin is performed by measuring the resin content of specimens cut from a sample both as received and with the surface resin removed. The amount of migrated resin can be reported several ways. In this study it was expressed as a percent of the total resin content (including the surface resin) of the specimen, using the following formulation:

Resin migration, percent = 
$$\frac{RC_1 - RC_2}{RC_1} \times 100$$

where

RC, = Resin content of the specimen as received, percent

'RC<sub>2</sub> = Resin content of the specimen after removal of the migrated surface resin, percent

The first study performed demonstrated the quantitative relationship between resin migration and winding tension, mandrel diameter, and roving resin content.

In this study, winding was performed on 3-, 8-, and 13-inch-diameter mandrels at 12, 18, and 24 pounds of tension, using roving having approximately 19 and 24 percent resin content. These samples consisted of circumferential windings only applied to a thickness of 0.06 inch. In addition, winding was performed on 3-inch-diameter mandrels at 6, 12, 18 and 24 pounds of tension, using roving having four different resin contents. These samples were standard 3-inch-diameter cylinders with a 0.065 inch-thick wall and a layer of fabric in the wall as described in the Appendix. The results of this study are shown graphically in Fig. 17.

Resin migration increased sharply with strand winding tension in the range from 6 to 24 pounds. Resin migration was greater in the 23 to 26 percent resin content range than in the 20 to 23 percent and 13 to 17 percent range, but less than in the 17 to 20 percent range. This appears to be inconsistent. However, the factor of flow, as affected by resin advancement, is probably having an influence on the resin migration. The study of resin flow was not included in this program, but certainly should be in future work.

The influence of part diameter on resin migration is shown in Fig. 18. Resin migration decreases rapidly as the diameter is increased. This is to be expected since the radial load or squeezing pressure of the tension applied to the strand is a function of the diameter of the mandrel and the amount of tension applied.

The relationship can be expressed in terms of the following equation for P, radial factor, T, winding tension, and D, mandrel diameter:

$$P = \frac{T}{D}$$

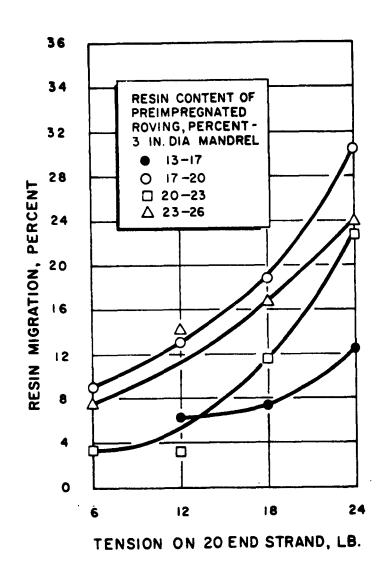


Figure 17. Effect of Tension and Roving Resin Content on Resin Migration

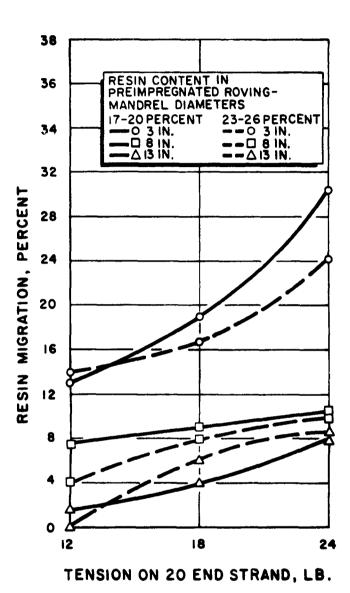


Figure 18. Effect of Mandrel Diameter and Roving Tension on Resin Migration

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Since resin migration is dependent upon both tension and diameter, it seems logical to correlate it to the load factor, P. This has been done in Fig. 19, using the data from Fig. 18. The load factor, P, for each tension and diameter is given in the chart below.

T 12 12 12 18 18 18 24 24 24

D 13 8 3 13 8 3 13 8 3

P = 
$$\frac{T}{B}$$
 0.92 1.5 4 1.38 2.25 6 1.85 3 8

It is evident in Fig. 19 that the resin migration and P relationship is practically a straight line. Deviations are probably caused by variations in the construction of the samples such as use of the fabric interply and variations in the resin content in the preimpregnated roving.

In another phase of this study, the effects of resin migration on the strength of filament-wound cylindrical samples was investigated. This was done by relating hoop tensile stress to resin content in the inner and outer layers of the samples. Thin-walled and thick-walled samples were fabricated. To prevent premature failure of cylindrical specimens during hydrostatic test, a layer of longitudinal filaments is placed in the center of the thickness of the wall. In the thin-walled (0.06° inch) samples (the same samples that were used in the first study)\*, a pre-impregnated fiberglass unidirectional fabric was used. This fabric had a resin content of approximately 20 to 22 percent which in some instances differed from the resin content of the preimpregnated roving used to wind

<sup>\*</sup> This is the standard 3-inch-diameter cylinder described in the Appendix for use in the hydrostatic burst test.

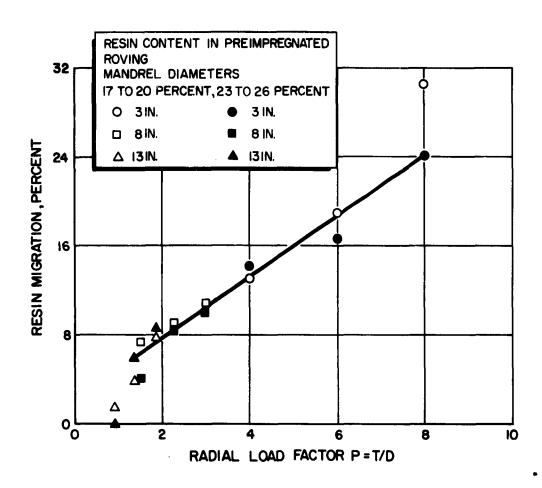


Figure 19. Effect of Radial Load on Resin Migration

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the samples. This caused the resin content percentage in some of the cured samples to be greater than in the roving, and in some of the cured samples to be less than in the roving. An adverse effect on the pattern of resin migration probably took place. Also, since the fabric was impregnated at a different time than the roving, it is expected that the resin in the fabric had a different flow characteristic. This also might have had an adverse effect on the pattern of resin migration.

To overcome these difficulties on the thick-walked (0.125-inch) samples, the same preimpregnated roving that was used for the circumferential wrapping was used for the longitudinal filaments. This was done by wrapping the preimpregnated roving on a flat plate to form a flat sheet of unidirectional fibers. A slight amount of heat was applied with an iron to make the resin sticky and to consolidate the roving into a sheet. The sheet was cut into smaller size pieces for placement in the filamentwound samples. These cylinders were constructed to simulate the construction of a pressure vessel. That is, the ratio of circumferential fibers to longitudinal filaments was two to one. Resin content was measured on the preimpregnated roving used for the circumferential windings and that used for the longitudinal filaments. The resin content was measured on the total wall thickness of the cylinders and on the inner and outer layers. Each sample cylinder was tested hydrostatically and the hoop tensile stress was calculated. These data are presented in Table 2. On a similar set of thin-walled cylinders (0.070 nominal), the resin content was measured on the inner and outer wall. Resin migration was calculated for the thin-walled and thick-walled samples. In the case of the thin-walled samples, migration was calculated as follows:

Resin Content Resin Content

Resin Migration = 

| Resin Content | In Inner Layer | In Inner Layer | Resin Content | In Total Wall Thickness | x | 100, % (1)



DATA ON RESIN CONTENT AND HOOP TENSILE STRENGT

FOR MIGRATION STUDIES OF THICK-WALLED 3-INCH-DIAMETER CYLINDERS

TABLE 2

Specimen No.	Circumferential Roving Resin Content, percent	Longitudinal Roving Resin Content, percent	Total Roving Resin Content, percent	Tension, pounds	Sample To Resin Cont percent
Low Resin Content					
568	21,2	20.3	21.0	12	21.1
569	20.7	20.3	20.6	12	18.6
570	19.4	20.6	19.8	12	18.4
571	21.1	20.6	20.9	18	16.1
572	20.9	20.6	20.8	18	18.6
573	20.8	21	20.8	` 18	18.3
574	20.8	21	20.8	24	18.3
575	20.5	21.5	20.9	24	19.9
576	21.1	21.5	21.2	24	18.2
High Resin Content					
577	21.9	25	22.9	12	22,0
578	21.8	25	22.9	12	22.6
579	265	23.7	25.6	12	21.8
580	23.2	23.7	23.3	18	21.6
581	22.2	23.1	22.5	18	20.1
582	22.1	23.1	22,4	18	21.1
583	20.8	22,4	21.4	24	19.1
534	27.1	22,7	25.6	24	22.5
585	27.6	22.4	25.9	24	22.

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Surface resin removed.

TABLE 2

ESIN CONTENT AND HOOP TENSILE STRENGTH
MIGRATION STUDIES OF THICK-WALLED
3-INCH-DIAMETER CYLINDERS

al Roving n Content, ercent	Tension, pounds	Sample Total Resin Content, percent*	Outer Layer Resin Content, percent*	Inner Layer Resin Content, percent	Ultimate Hoop Tensile Strength, psi
21.0	12	21.1	19.7	15.6	247,000
20.6	12	18.6	20.1	15.8	246.000
19.8	12	18.4	20.6	14.8	244,000
20.9	18	16.1	16.5	15.0	239,000
20.8	18	18.6	19.8	14.9	240,000
20.8	, 18	18.3	20.6	16.2	252,000
20.8	24	18.3	22.5	14.3	249,000
20.9	24	19.9	21.8	14.6	235,000
21.2	24	18.2	20.8	16.2	248,000
22.9	12	22.0	21.4	18.5	215,000
22.9	12	22.6	20.6	18,8	221,000
25.6	12	21.8	21,6	17.4	237,000
23.3	18	21,6	22.1	16.0	218,000
22.5	18	20.5	21.2	15.5	212,000
22 , 4	18	21.2	21.8	15.6	226,000
21.4	24	19.4	19.5	18.4	238,000
25.6	24	22.9	25,5	17.1	206,000
25.9	24	22.8	24.4	17.2	213,000



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Resin content in the outer layer was measured, with the migrated surface resin removed. Resin migration was calculated with two methods for the thick-walled cylinders. One procedure was as above, the other was as follows:

Resin Migration = 
$$\frac{\frac{\text{Resin Content}}{\text{in Roving}} - \frac{\text{Resin Content}}{\text{in Total Wall}} \times 100, \%$$
 (2)

The resin content in the total wall of the cylinder was measured, with the migrated surface resin removed. The roving resin content was determined on the roving used for longitudinal and on that used for circumferential filaments. Since one-third of the sample was made of longitudinal fibers and two-thirds circumferential, the total resin content was determined as follows:

1/3 Resin Content (longitudinals) + 2/3 Resin Content (circumferentials) = Total Resin Content of Roving Used to Make Sample

The resin migration data for the thick-walled and thin-walled cylinders are presented in Table 3 and 4, respectively.

TABLE 3

# RELATIONSHIP OF TENSION AND ROVING RESIN CONTENT TO RESIN MIGRATION, 3-INCH-DIAMETER THIN-WALLED (0.060-INCH-THICK) CYLINDERS

Specimen No.	Resin Content In Roving, percent	Tension, pounds	Resin Content**, percent	Migration*, percent	Hoop Tensile Strength, psi
514	17 to 20	6	18.2	10.4	253
517	17 to 20	12	16.4	7.3	260
520	17 to 20	18	15.4	7.2	251
524	17 to 20	24	14.6	14.4	236
509	13 to 17	18	16.2	6.2	260
532	20 to 23	18	19.9	19.6	251
545	23 to 26	18	18.8	11.2	216
	1	1	1	1	1

<sup>\*</sup> Resin Content Resin Content
Outer Layer Inner Layer
Resin Content Total Wall (Eq. 1 on p. 54)

<sup>\*\*</sup> Total wall, surface resin removed

TABLE 4

RELATIONSHIP OF TENSION AND ROVING RESIN CONTENT
TO RESIN MIGRATION (3-INCH-DIAMETER CYLINDERS
WITH 0.120-INCH NOMINAL WALL THICKNESS)

Roving Resin Content 18 to 22 Percent	Migration**,	3.9 1.3 Average 	7.3 Average 8.9 Average 5.3	9.3 10.5 Average 12.0
	Specimen No.	577 578 579	580 581 582	583 584 585
	Migration*, percent	13.2 8.0 Average 8.0 15.5 19.3	27.8 Average 27.8 28.3 29.2	 26.7 Average 31.5
	Migration**, percent	 10.1 Average 7.1	12.0 10.6 Average 11.5	12.0 Average 13.1 14.1
	Specimen No.	568 569 570	571 572 573	574 575 576
18 t	Migration*, percent	4 Average 1 24.7	4 Average 25.1	7 Average 1 35.3
		19.4 25.1 31.5	26.4 23.9	44.7 36.1 25.2
	Tension, pounds	12 12 12	18 18 18	**************************************

\* Migration = Outer Layer Inner Layer (Eq. 1 on p. 54)

Resin Content Total Wall

\*\*Migration = In Roving In Total Wall (Eq. 2 on p. 56)

Resin Content in Roving

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Some of the values of migration in Table 4 are not included in the average value presented. This has been done because examination of the data in Table 2 for the specimen involved indicates that the data are not reliable. An example involves the value of migration calculated using Eq. 2 for specimen 568. In using the data from Table 2, the resin migration value would be 0 and out of line with values determined for specimens 569 and 570. This happens because the resin content in the cured sample (taken with the migrated surface resin removed) is the same as the value for resin content in the roving used. This seems incongruous since, if the migrated surface resin had been properly removed, the resin content in the sample then must of necessity be lower than that in the roving used. The specimen therefore is assumed to have been improperly treated and the value obtained inaccurate, and it should not be included with the others in the series.

It is apparent with both the thick- and thin-walled cylinders that the resin migrates to the surface. This reduces the resin content in the inner layers and increases the resin content in the outer layers (even when the excess surface resin has been removed). It is possible for the outer layers to take on resin so that the resulting content, by percent, is greater than the percentage in the preimpregnated roving used to fabricate the sample. It is also noted that the values of migration for the thick-walled cylinders are greater than for the thin-walled cylinders. This is to be expected since a greater radial pressure is built up in a thick wall than in a thin wall.

In the thick-walled samples, it is apparent that the migration of resin from the inner layers to the outer layers increases as the tension on the roving strand is increased. Although there can be a significant difference in resin content between the inner and outer layers, only a relatively low

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percentage of the total resin content is squeezed to the surface. This is indicated by the lower values for migration calculated by Eq. 2 than those calculated by Eq. 1.

The data in Table 4 indicate that the resin migration does not increase with resin content in the roving, but actually decreases on a percentage basis. This would indicate that the amount of resin which migrates to the surface is constant, and consequently the percentage is less for higher resin content roving. Resin migration occurs because the tension in the roving creates a radial pressure, analogous to applying a vacuum bag over the laminate, which squeezes the resin to the surface. This is enhanced during cure when the resin softens from the heat and flows before gellation occurs. The flow of resin allows relaxation of the tension in the roving which reduces the radial pressure and the tendency for additional migration. The migration limit appears to depend not on the resin content but on the radial pressure available.

The relationship between resin migration and hoop tensile stress is not evident in the thin-walled cylinders, nor is the pattern of inner-to-outer-layer migration relative to tension or roving resin content as evident as in the thick-walled cylinders. The relationships are probably clouded by the use of the layer of preimpregnated fabric, as discussed in detail above.

The relationship between resin migration and tensile strength in the thick-walled cylinders is not clearly evident. It may be clouded by the variations and inconsistencies in the construction of the samples. Preimpregnated roving was used for the longitudinals in order to have a material of the same resin content and flow characteristic as in the circumferential windings. Although material for the longitudinals was

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from the same batch as that used on the circumferential windings, it was not possible to match the resin content. The manufacturer's tolerance on the resin content of the preimpregnated roving is plus or minus 2 percent. In the range selected for samples 577 to 585 (Table 2) this allows a variation from 22 to 26. Actual measurements ranged from 20.8 to 27.6 percent. This variation was not predicted; consequently, material having a wide range of resin content inadvertently was used in some samples.

Examples are samples 577 and 578 where the resin content in the longitudinals is 25 percent compared to slightly less than 22 percent for the resin content in the roving for the circumferential windings. This accounts for the peculiar situation with these samples where the percent resin content in the total wall of the cured sample is greater than the percent content in the outer layer (migrated surface resin removed in both instances).

An examination of the data in Table 2 reiterates the relationship of resin content, both in the preimpregnated roving and in the cured sample, to hoop tensile strength described earlier in the report. The average strength of specimens 568 to 576 is higher than the average strength of specimens 577 to 585. The resin content values are in inverse ratio, as expected.

Another example of this phenomenon is illustrated by specimen 583. This specimen was made as a part of the group, numbers 583,584 and 585, wound with 24 pounds of tension, using high-resin-content roving. This specimen has the highest tensile strength of the three specimens and, in fact, of many of the specimens included in the group from 577 to 585 made with high-resin-content roving. At the same time, the roving resin content of 21.4 percent and the sample resin content of 19.4 percent are the lowest

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of any specimen in the high-resin-content group."

The specimen also seems out of place as indicated by the resin migration value of 5.7 percent, calculated by Eq. 1. This value compares to 36.7 and 31.5 for specimens 584 and 585, respectively. Because it was so badly out of line, it was left out of Table 4. The roving resin content and sample resin content values of 21.4 and 19.4 percent for specimen 583 would indicate that this specimen actually belongs in the 568 to 576 group. Because it was wound at 24 pounds tension, it should fit in with specimens 574, 575 and 576. However, the inner- to outer-layer resin migration value of 5.7 percent (calculated using Eq. 1) does not match with the 35.3 percent average value for specimens 574 through 576. This same situation existed when 583 was compared with 584 and 585. The discrepancy involved would seem to reflect a difference in resin flow in the materials used.

Resin flow is a significant property of preimpregnated roving which influences resin migration. In this respect it also influences strength properties. This is probably more significant in larger diameter filament—wound structures than in 3-inch-diameter cylinders. The influence of resin flow was not included in this program but warrants study, along with additional investigation of resin migration.

## Tackiness and Volatile Content

The purpose of this study was to determine the relationship of tackiness in the preimpregnated roving to volatile content, resin content, processing parameters, and properties of the cured laminate. The first step in the investigation was to develop a suitable test for measuring tackiness.

Several different procedures were studied for evaluating tackiness in preimpregnated roving. One method measured the tackiness of roving by allowing a steel cylinder to roll down an inclined ramp along two parallel strands of roving spaced apart on a horizontal surface. Tackiness was measured in terms of the distance the roller moved along the strands of roving i.e., the greater the tackiness, the more the motion of the roller was impeded and the shorter the distance it rolled before coming to rest.

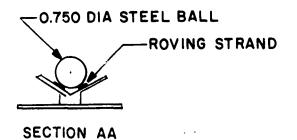
The test procedure was found to have a number of serious flaws. High spots or slight twists in the roving prevented the roller from making complete contact with the tacky surface of the strand. Because of this factor, it was difficult to obtain reproducible test results. In addition, it was intended that roving strands of various widths (20 ends, 60 ends, etc.) would have to be tested. Each width would impose a different set of test conditions because of the variation in areas involved. For these reasons, tackiness testing by this method was not continued.

In another test, strands of preimpregnated roving, 12 in. long, were placed on a chrome metal mirror plate and heated for 10 minutes in an oven at 20 F. The strands were then pressed firmly against the polished metal surface by five passes of a 2-pound roller. The plate was placed at a 45-degree angle, and the time in seconds required to peel a 10-inch length of strand from the plate by the pull of a 100-gram weight was taken by stopwatch. This test gave consistent and reproducible results for several lots of roving. However, several other lots of roving strands failed to adhere to the surface of the metal plate. This result may be due to resin advancement.

In another method, the time required to peel a 2-inch length of strand from the surface of a spool of roving by the pull of a 100-gram weight was taken by stopwatch. The time required was the measure of tackiness. This method gave consistent results for most lots of roving, although the tendency of individual filaments to detach from the roving strand affected the rate of peel in a number of cases.

In still another method, the tackiness tester consisted of a flexible band of steel 20-inches long by 3/8-inch wide by 0.015-inch thick. The band was placed over a horizontal spool of roving in the spool box of a winding machine. A weight of 2 kilograms was hung on one end of the steel strap; the other end of the strap was connected to a spring balance. As the spool of roving turned at a constant rate, the strip of steel acted as a brake band, and the pull of the band against the surface of the roving spool was registered on the spring balance. This, in effect, was a measure of the friction factor of the roving surface which was related to its tackiness. This method appeared to work fairly well, although more tests are needed to complete the evaluation.

The method that was selected is described below. The equipment consists of an inclined ramp and horizontal track as shown in Fig. 20 and 21. The ramp, inclined at 10 degrees, is approximately 18 inches long. The track is 60 inches long. Both are made of V-shaped aluminum extrusion. The strands of roving are placed on the track about 3/8 inch from the point of the V and stretched tightly along the track by weights (4 pounds for each 20 end in each strand). A 3/4-inch-diameter steel ball is placed at a specified height on the ramp and allowed to roll down the ramp and along the track in contact with the roving. The distance the ball travels along the track is the measure of tackiness. The nature of the test is such that a low number indicates more tack than a higher number.



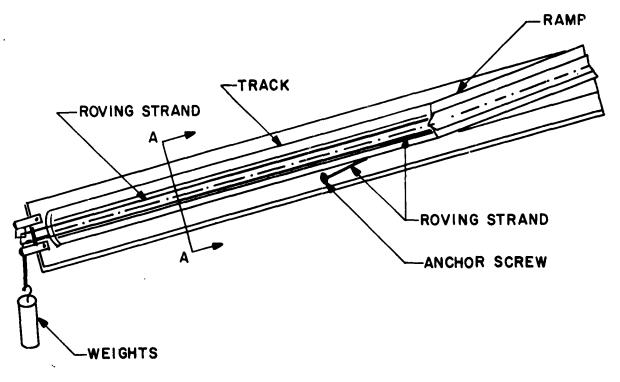


Figure 20. Tackiness Tester for Preimpregnated Roving



Tackiness Testing Device 21.

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Measurements were made on a selected group of spools of preimpregnated roving to establish a base for comparison. These spools were fresh material (stored at deep-freeze temperature since the day of manufacture). Table 5 gives results. The first digit of the value of tackiness is the distance along the ramp the ball rolled. The second number is the distance along the track and is the average of five rolls. The values reported in Table 5 are listed in order of decreasing tackiness, from left to right. The differences reported were readily detected by "finger feel" and are in agreement with this subjective test.

TABLE 5
TACKINESS TEST VALUES

Spool No.	8	10	24	28	35	44	46
Tackiness Value	8-27	8-28.5	8-29	8-31	8-32	8-35	8-36
Temperature, F	84	84	85	81	84	82	84

The procedure used for the tests above and all subsequent tests consisted of rolling the ball six times over each set of specimens. The ball was cleaned with solvent before each pass. The average value of the distance traveled for the last five passes is reported. In this test it was found that the ball would iron out irregularities in the strand of fresh material on the first pass over the roving, then, on the second pass, travel considerably farther along the track. The ball rolls a greater distance each time on subsequent passes, but the increments are progressively smaller. This is why zero-in roll is used.

In another experiment, an attempt was made to relate storage of preimpregnated roving at room temperature to change in tackiness and volatile content. Samples of roving were removed from the outside of the spool each day, and measurements of each property made. In essence, measurements were made on surface material only. An analysis of Fig. 22 shows that the volatile content drops, but no trend of tackiness is discernible by this tackiness test method, although reduction in tackiness is discernible by feeling the roving.

The erratic results indicated in Fig. 22 may be accounted for by the explanation that as the roving ages it becomes hard and rigid. Small wrinkles are not relieved when the roving is placed on the track and tension is applied by the 4-pound weights. These wrinkles retard the travel of the steel ball, especially near the end of its roll along the track. The older the preimpregnated roving is, the greater this influence which could account for the apparent reversal of measured tackiness value.

Experiments have shown that the test is useful only on fresh or soft, pliable roving which has a degree of tackiness that can be felt when the roving is gently squeezed between the fingers. This was indicated by the tackiness values determined on the material used in the tension and resin content study which was reported in Table 6, along with resin content, volatile content, and manufacturers' lot number of preimpregnated roving. The values correlate well with the finger-feel test and have a fairly narrow spread within each lot. However, no relationship with volatile content or resin content is discernible.

The nature of the tackiness test is such that the measurement range may be extended in either direction, low or high tackiness, by the distance the ball is allowed to roll down the inclined plane. This is comparable to changing scales by changing weights on a Rockwell hardness test. However, the distance most often used is 8 inches up the inclined ramp.

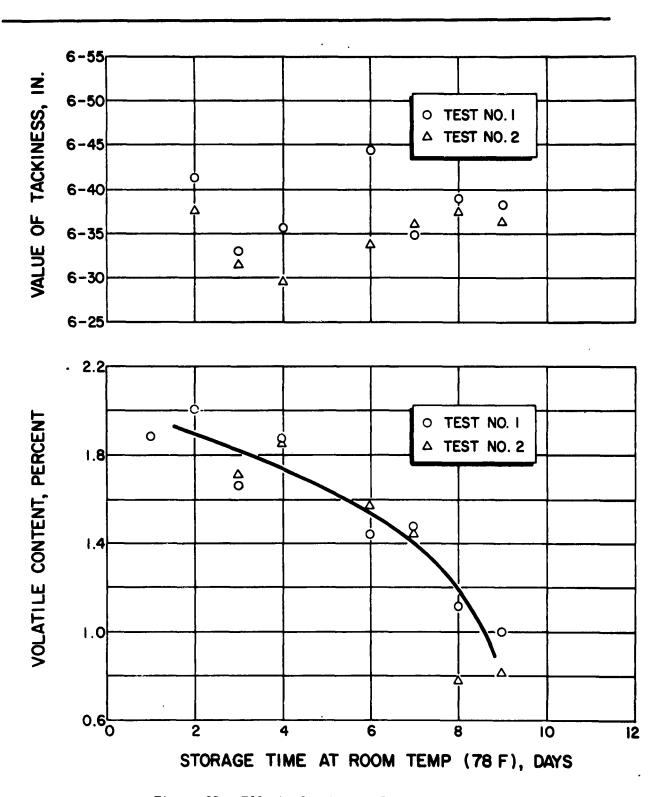


Figure 22. Effect of Aging on Tackiness and Volatile Content

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TABLE 6

PROPERTY DATA OF ROVING USED FOR TENSION
AND RESIN CONTENT STUDY

Spool	Sample Taken From Specimen No.	Volatile Content, percent	Resin Content, percent	Tackiness Test Value
696-2 696-2 696-3 696-4 696-10	501 502 503 505 508 511	1.43 2.74 2.76 1.68 1.68 1.67	13.7 14.8 15.3 16.2 16.9 17.4	8-45.4 8-42.0 8-38.9
646-48	513	1.87	18.3	8-37.7
646-48	514	1.90	18.4	8-35.0
646-48	515	1.68	19.6	8-34.2
646-53	520	1.90	18.9	8-31.0
690-2	525	2.04	21.0	8-42.6
690-2	528	1.85	22.4	8-49.4
690-3	529	1.66	22.4	8-39.0
690-3	530	1.70	19.6	8-37.2
690-4	532	1.39	24.6	
690-7	534	2.56	22.2	8-50.7
589-1	537	1.57	24.2	8-27.7
589-1	539	1.55	25.0	8-20.9
589-2	541	1.31	25.2	8-21.9
589-2	544	1.43	24.0	8-28.1
589-3	546	1.52	25.4	8-28.3

The temperature at which the test is performed is significant. Measurements reported in this paper were made at 75 to 78 F. The tackiness of the roving will increase with temperature. A very interesting observation made during development of this test was that materials which had significantly different values of tackiness at 77 Fhad practically the same value of tackiness at 12 F.

The next step, with the tackiness test method developed, was to relate the tackiness of preimpregnated roving to process parameters.

For this purpose, a study was undertaken to determine the influence of preimpregnated roving tackiness on the ability to wind an unstable pattern.

The work was performed on a mandrel which measured 18 inches in diameter by
24 inches long. The pattern chosen was a low-angle helical or pole-to-pole
pattern at an angle of 20 degrees to the longitudinal axis. The winding was
performed, using a single strand of 20-end preimpregnated roving of the same
type used throughout the program. The pattern of application to the mandrel
was sequential, with each strand spaced equally, parallel to the preceding
strand, and spaced with approximately a 30-percent overlap.

Trial windings of the roving were placed parallel to each other on the mandrel, with a progressively increasing polar diameter of the wrap at one end and decreasing diameter at the other end. The winding angle for the cylindrical portion was kept at 20 degrees to the axis (Fig. 23). When the polar diameters at each end of the winding were equal, the diameter was 8.4 inches at each end. Tests for slippage were made by increasing the diameter at the top of the mandrel by 2.4-inch increments until slippage occurred.

These tests were made using preimpregnated roving of two levels of tackiness, as measured on the rolling ball tackiness tester. The low-tack spool, No. 696-8, had a tack value of 8->60. The high-tack spool No. 646-57, had a tack

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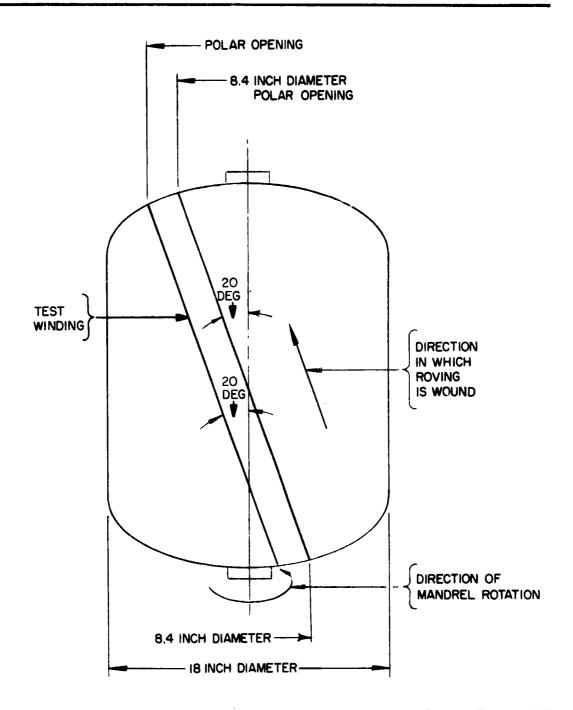


Figure 23. Winding Pattern for Study of the Effect of Preimpregnated Roving Tackiness on the Ability to Wind an Unstable Pattern

value of 8- 32.4. To convey some meanings to these tackiness values, the low value (high-tack) material is slightly sticky to the touch when squeezed between the fingers, and the high value (low-tack) material is very "dry" to the touch.

The first part of the investigation consisted of performing the slip test winding on a bare mandrel. This mandrel was made of plaster, sealed with a cellulose acetate plastic film and coated with a nonslippery wax mold release. A few xraps of the low-tack preimpregnated roving were wound on the mandrel. If no slip occurred, these wraps were removed from the mandrel and another wrap was made at a larger polar diameter at the top (smaller at the bottom). This procedure was continued until slip occurred.

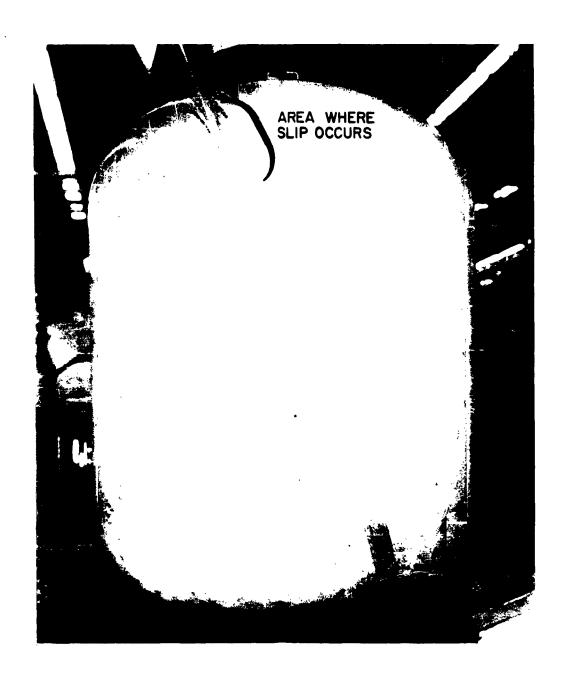
The position on the mandrel where this happened is shown in Fig. 24. A slight amount of slip was considered to have occurred when the strand being applied moved out of position after contacting the mandrel to increase the spacing relative to the adjacent strand, this occurred on the bare mandrel at a polar diameter at the top of 9.6 inches. Severe slip was considered to have occurred when the wrapping slipped compactely off the mandrel. With low-tack roving used, this occurred at a polar diameter of 12 inches.

When slipping occurred with the low-tack material, use of the high-tack material was begun. The procedure described was followed until slip occurred. Slight slipping occurred, with this material, at a polar diameter of 14.4 inches, (Fig. 25).

For the second phase of this investigation, the mandrel was covered with a "base wrap" of low-tack preimpregnated roving. This was applied as a pole-to pole wrap to contact the polar bosses. Trial windings of low-tack roving were then placed over this base wrap in the same manner as application to

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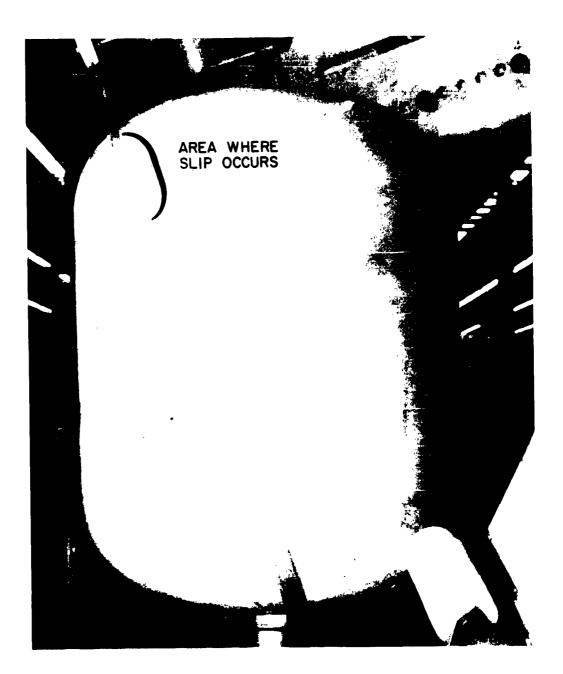
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Figure 24. Winding Low-Tack Preimpregnated Roving on a Bare Mandrel

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6930-2/8/62-2A

Figure 25. Winding High-Tack Preimpregnated Roving on a Bare Mandrel

the bare mandrel had been made. The experimental wrapping setup is shown in Fig. 26.

When the low-tack material began to slip, the experiment was continued with high-tack material over the base wrap. Upon completion the base wrap of low-tack roving was removed, and a wrapping of high-tack preimpregnated roving applied. The slip experiments were repeated, using both low- and high-tack roving. The polar diameter values at which slipping occurred are given in Table 7.

The data in Table 7 indicate that there is almost the same tendency for the strand to slip when wrapping on a base wrap of preimpregnated roving as when wrapping on the bare mandrel. This condition was not anticipated. It was thought that there would be considerably more resistance to slip when wrapping preimpregnated roving over roving because of the pressure-sensitive cohesive action of the resin.

An explanation for the above experiments may be that since the roving strands in the base wrap overlap, the wrap has a stepped, or rough, surface condition. The test winding placed over this rough-surfaced base wrap makes point contact with the high spots of the surface only to provide a smaller area of contact than when applying roving to the bare mandrel. Although there may be more stickiness in the roving to roving contact, this is offset by the lower area of contact, resulting in less resistance to slip than anticipated.

This study has shown conclusively that it is possible to wrap a more unstable pattern with high-tack preimpregnated roving than low-tack roving. In addition, it has been shown that preimpregnated roving can be used to wind a polar opening of nearly 70 percent of the case diameter, using a



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Figure 26. Winding Preimpregnated Roving Over a Wrap of Preimpregnated Roving

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TABLE 7

## THE INFLUENCE OF TACKINESS ON THE ABILITY TO WIND AN UNSTABLE PATTERN

I	Polar Diameter of Test Winding on ABL* Mandrel, Inches and Percent of Mandrel Diameter.				
Low Tack Rov	Low-Tack Roving, Tack Value 8- >60 High-Tack Roving, Tack Value 8- 32.				
Wind On Bare Mandrel	Wind On Low-Tack Wrap	Wind On High-Tack Wrap	Wind On Bare Mandrel	Wind On Low-Tack Wrap	Wind On High-Tack Wrap
8.4 inches 47 percent No Slip		8.4 inches 47 percent No Slip			
1 *	9.6 inches 53 percent No Slip	10.2 inches 57 percent Slight Slip	53 percent		
	12 inches 67 percent Slip	12.6 inches 70 percent Slip		12 inches 67 percent No Slip	12.6 inches 70 percent No Slip
			80 percent	14.4 inches 80 percent Slight Slip	

<sup>\*18</sup>-inch-diameter x 24 inches long. Test winding applied at constant 20-degree angle to longitudinal axis.

nongeodesic or unstable pattern. This study also indicates the potential usefulness of the ability to quantitatively measure the tackiness of preimpregnated roving.

It should be noted that the polar opening at which slip will occur is dependent upon the end dome configuration and the length to diameter ratio of the case. These factors must be considered when applying the data in Table 7 to a specific example.

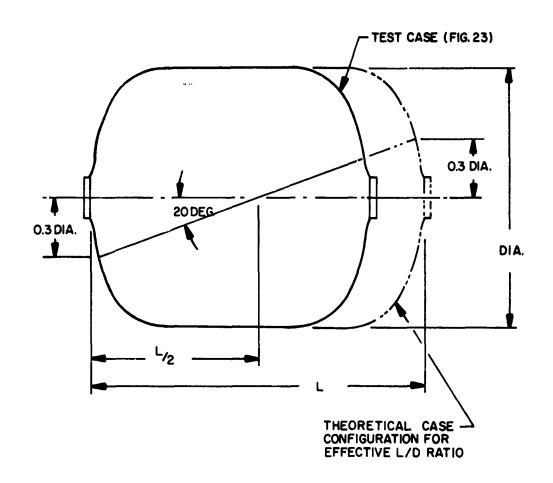
To illustrate: The data presented in Table 7 were collected using highand low-tack roving. On a bare mandrel and with low-tack roving, slight slip occurred at a polar opening of 53 percent of the case diameter. With high-tack roving, slight slip occurred at 80 percent of the case diameter. Therefore, it can be assumed that medium-tack roving would have slight slip at 60 to 70 percent.

Figure 23 shows that the roving was placed on the mandrel at a 20-degree angle to the axis. Contrary to the description in Fig. 23, it will now be assumed, in the example under consideration, that the polar wrap openings are of equal diameter, that the openings are 60 percent of the diameter and the roving is placed at a 20-degree angle. This results in an effective length to diameter ratio for the case of 1.65. The calculation is shown in Fig. 27.

If roving of the same tackiness were used on a mandrel with a similar end dome shape, but with a greater length to diameter ratio, and the roving was wound with a polar opening of 60 percent of the diameter, the roving would be expected to slip. Although all other conditions are constant, the greater length to diameter ratio has introduced a condition of greater instability. This was exemplified during fabrication

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Tan 20 degrees = 
$$\frac{0.3 \text{ D}}{\text{L}/2}$$
 or Tan 20 degrees =  $\frac{0.6 \text{ D}}{\text{L}}$ 

$$\therefore \frac{\text{L}}{0.6 \text{ D}} = \frac{1}{\text{Tan 20 degrees}} \quad \text{then } \frac{\text{L}}{\text{D}} = \frac{0.6}{\text{Tan 20 degrees}}$$

$$\frac{\text{L}}{\text{D}} = 1.65$$

Figure 27. Calculation of Effective L/D Ratio for Tackiness Study

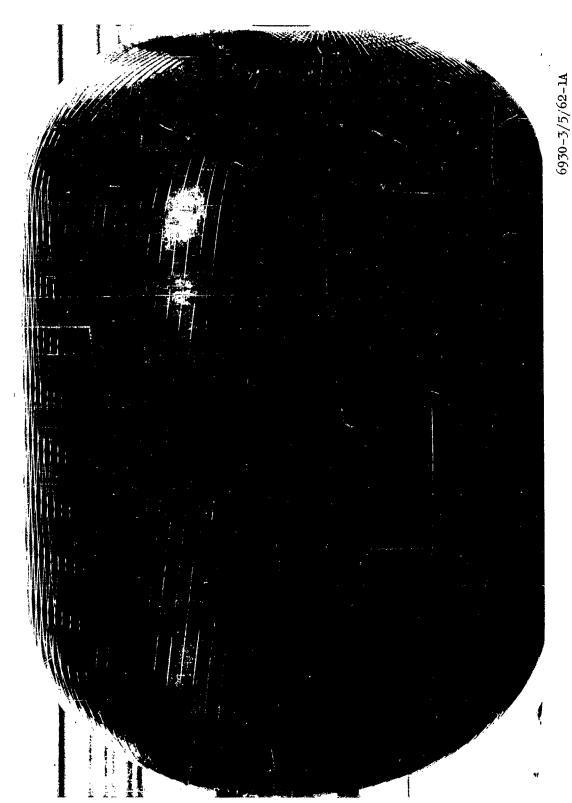
of a case on another program when a mandrel having a L/D = 1.87 was wrapped with a polar opening of 60 percent of the diameter. Severe slipping of the preimpregnated roving occurred. In this instance it would have been possible, by using the above analysis, to predict the slippage condition and specify remedial measures such as use of very tacky roving.

Another example of the significance of tackiness was demonstrated during checkout of the numerically tape-controlled winding machine. A polar wrapping pattern was being applied to an 18-inch-diameter by 24-inch-long mandrel. The purpose was to check the accuracy of spacing of the strand. To do this, a mercerized cotton thread was used (Fig. 28 and 29). The spacing was not uniform over the end domes. It was noticed that the thread would slip as it traversed the buildup section around the polar opening (Fig. 29). Each strand subsequently was taped in place as it traversed the buildup area. With no slippage, the spacing of the thread was uniform. In essence, use of the tape was the same as creating tack on the thread to prevent it from slipping away from the true laydown path.

The same situation occurred when preimpregnated roving was substituted for the cotton thread. The initial tests were made with low-tack material, and the strand spacing was irregular on the end domes for the same reason as with the cotton thread. No slip occurred after switching to tacky roving, and the strand spacing was uniform. From this and subsequent experiences it has been determined that the use of pliable, tacky preimpregnated roving is a necessity when winding low-angle helical patterns (polar pattern) on the NTC machine. It will be possible, with development of the rolling ball tackiness tester, to specify a minimum useful level of tackiness for this application.

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Over-all View of Test Pattern Performed by NTC Machine Using Pattern Thread Figure 28.

End View of Test Pattern Performed by NTC Machine Using Cotton Thread

Figure 29.



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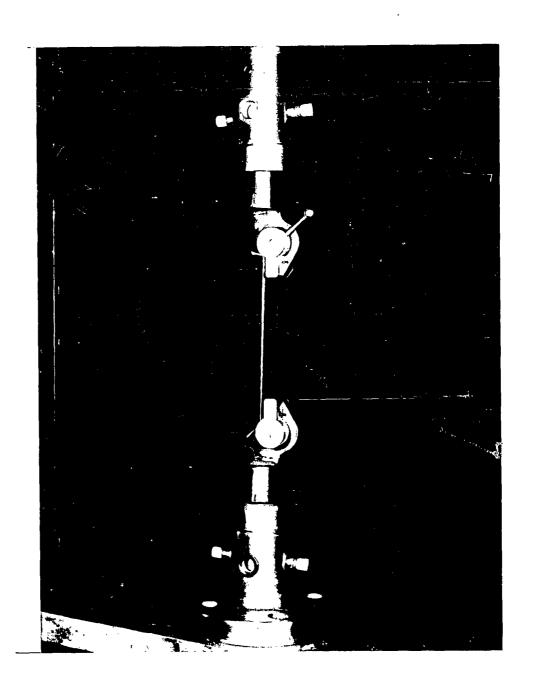


## STRAND TENSILE STRENGTH

Quality control standards, particularly with reference to receiving inspection and inspection are dependent upon standard test procedures. It was intended that the tensile strength of the uncured strand of preimpregnated roving be included as a standard in the specification to be written, if found it be significant. Test procedures then were developed, and a program to determine test conditions and standard values was undertaken.

The testing apparatus whosen was that shown in Fig. 30, in which roving was wrapped around Scott spool clamps to prevent the possibility of premature failure from stress acceptuation that would occur if conventional tensile specimen jaws were used. Testing was performed in an Instron tensile testing machine. The load was applied at a prosphead travel of I much a minute. A series of tests or 200-end strand was made to determine whether strand length was signed, and. The data are presented in Table 8 All subsequent testing was based on these tests and was performed on 6- inch-long samples (a length convenient to use).

If this testwere to be useful, then measuremeans made on samples taken from the outer portion of the spool of preimpregnaned roving must represent the quality throughout has spool. To determine this, measurements were made on samples taken from the raper, middle, and outer layer of a single spool. In appears from the lake presented in Table 9 that samples taken from the cultide of a spool afford a rectable estimate of swand beasile strength throughout the spool. This is probably true since a spool of preimpregnated verify without spool brows has to be made from only one spool of the regimes roving. However, madismables of his non-clusion will require resents of many more spools of preimpregnated roving.



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Figure 30. Fixture for Strand Tensile Test

Since the strand tensile strength test was to be performed on the resin-loaded roving, it was important to know whether resin content would influence the strand strength. In this test, the effect of resin content of the roving upon the breaking strength of a 20-end strand was measured. The data are presented in Table 10 Values for the low-and medium- resin-content samples are in the same range as the values previously reported in Table 9 Values in the high-resin-content range are much lower and are more scattered. This cannot be accounted for by the higher resin content, as the difference between the two higher-resin-content samples is only 7 percent of the nominal resin content. It is possible that roving used to make this "prepreg" had considerable catenary. Another sample of material from the same lot was tested to contirm the previous results. The following data were obtained using the same test procedure.

Spool No.

**587** .

Resin content, percent

Tensile strength, pounds

82 4 (average of 10 specimens)

During this test the strand broke in a progressive manner, i.e., not all of the 20 ends failed simultaneously. The peak load during the test was reached with some of the filaments intact. This is similar to the type of failure experienced on spool No. 589-1. The evidence is strong that catenary was present in the glass roving used to make this batch of pre-impregnated roving.

Another test of the tensile strength of preimpregnated, uncured strand was made. In this test, the effect of room temperature aging was measured. The roving sample was a 20-end strand 6-inches long. It was tested by the same method as other samples in this series. Measurement was made of

tensile strength, volatile content, and resin content on the material, as received, after 1, 2, and 3 weeks at laboratory ambient conditions. All material was taken sequentially from the same spool of roving. The results are presented in Table 11. It is possible that the increased strength after one week of aging is due to curing of the resin. The progression of cure is indicated by the loss in tackiness and stiffening of the strand. However, the strength reduction after 2 and 3 weeks would seem to counter this theory. Consequently, no explanation has been determined.

In spite of the unknowns encountered in the last experiment, the foregoing study indicates the potential usefulness of the strand tensile strength test for quality control. Eventual use of this test will depend on the ability to relate tensile strength of the uncured strand to performance in the filament—wound structure.

TABLE 8

STRAND TENSILE STRENGTHS OF UNCURED PREIMPREGNATED ROVING\*

12-inch Long Strand, pounds	6-inch Long Strand, pounds	3-inch Long Strand, pounds
97	104	106
105	109	105
98	104	109
108	106	109
<u>103</u>	<u>105</u>	106
5 511 : 102	5 528 = 105	5 535 = 107

<sup>&</sup>quot;20-end E glass with 801 sizing

TABLE 9 TENSILE STRENGTH OF UNCURED PREIMPREGNATED ROVING FROM SINGLE SPOOL\*

Sample** No.	Outer Layer Strength, pounds	Middle Layer Strength, pounds	Inner Layer Strength, pounds
1	109	122	115
2	106	111	118
3	101	97	112
4	119.5	114	118
5	105.5	85	121.5
6	101.5	108.5	106
7	114.5	105	111
8	112	105	120
9	109	112	107
10	108	112	107
χ̈̈́	108.6	107.2	113.6
s	5.4	9.1	4.2
v	5.0	8.5	3.7

 $\overline{X}$  = average S = standard deviation V = coefficient of variation =  $\frac{S}{\overline{X}}$  X 100

\*Length of samples, 6 inches

\*\*20-end E glass with 801 sizing

TABLE 10
TENSILE STRENGTH OF UNCURED PREIMPREGNATED ROVING\*

Sample	Spool 696-5 Strength, pounds	Spool 690-4 Strength, pounds	Spool 589-1 Strength, pounds
1	109	108	62.5
2	87	97.5	62
3	99	108	89
4	102	107.5	68
5	97	109	85
6	92	104	87
7	96	106	71
8	96	98	76
9	99	101	85
10	97	104	83
X (average)	97.4	104.3	76.9
s (standard duration)	5.5	4.0	9.4
v (coefficient of variation)	5.6	3.8	12.2
Resin content, percent	17.3	25.2	27.0

<sup>\*20-</sup>end E glass with 801 sizing

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TABLE 11 TENSILE STRENGTH OF UNCURED PREIMPREGNATED ROVING STORED AT ROOM TEMPERATURE\*

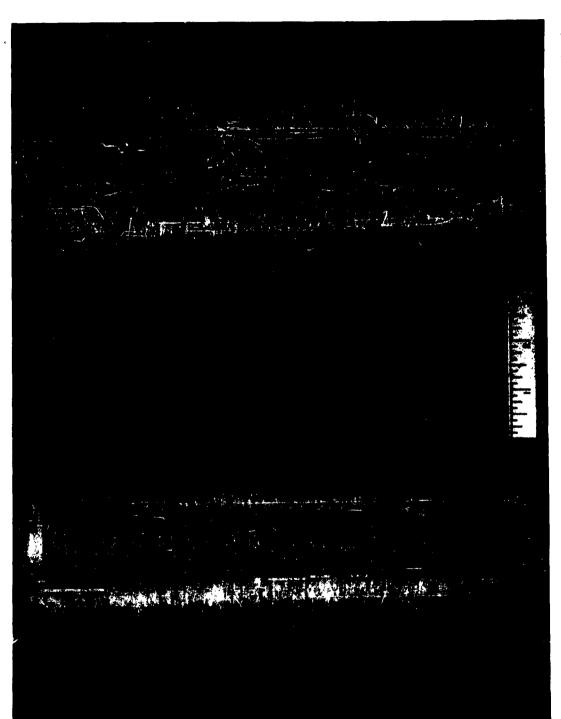
Sample	Tensile Strength, pounds	Volatile Content, percent	Resin Content, percent
As Received		1.91	20.0
No. 1	89.0		
No. 2	77.5		
No. 3	90.5		1
No. 4	86.0		
No. 5	89.5		[
Average	86.5		
After 1 Week Aging		1.43	19.2
No. 1	113.0		
No. 2	109.0		
No. 3	110.0		į
No. 4	97.0	}	
No. 5	108.0		
Average	107.4		!
After 2 Weeks Aging		1.73	19.3
No. 1	88.0		1
No. 2	72.0	İ	1
No. 3	77.5		I
No. 4	96.5		
No. 5	91.0		}
Average	85.0	!	<u>.</u>
After 3 Weeks Aging	, ,	**	**
No. 1	93.0	:	
No. 2	92.0	i	
No. 3	86.0	·	
No. 4	78.0		
No. 5	90.0		1
Average	87.8		

<sup>\*20-</sup>end E glass with 801 sizing
\*\*Not determined because of poor sample condition

## Packaging Study

In this study, the purpose was to compare two different package designs and the best method of applying tension to the strand. The two package designs were a parallel or straight wind (left side of Fig. 31) and a 75-degree way wind (right side of Fig. 31). In this investigation, the preimpregnated roving used was the same as that which had been used throughout the program. Two methods of applying tension during winding were used. One method consisted of resisting the rotation of the spool of roving. This is described as applying tension at the spool or package. In the other method, the tension was applied to the strand by passing it over a system of brakes (Fig. 32).

By using these two methods of applying tension, 3-inch-diameter cylindrical samples were fabricated, using material from each package shown in Fig. 31. The cylindrical samples were tested hydrostatically at room temperature, and the ultimate hoop tensile strength was calculated: the purpose was to determine what effect applying tension at the package, compared with applying tension on the strand, would have on the tendency to degrade the fiberglass roving. Any degradation would be expected to show up in reduced strength in a filament-wound sample. Results are shown in Table 12. The data indicate that some degradation occurs in the strand when tension is applied at the spool in the case of the waywind package. No test of cylinders made by applying tension at the parallel-wound package was made because of shortage of material. A repeat test was made on the way-wind package to verify the results obtained in the initial test. This was done because, in the original experiment, the samples in which the tension was applied to the package were fabricated before those on which the tension was applied only to the strand. Since all samples were fabricated from the same spool



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Examples of Two Different Types of Preimpregnated Roving Packages Figure 31.

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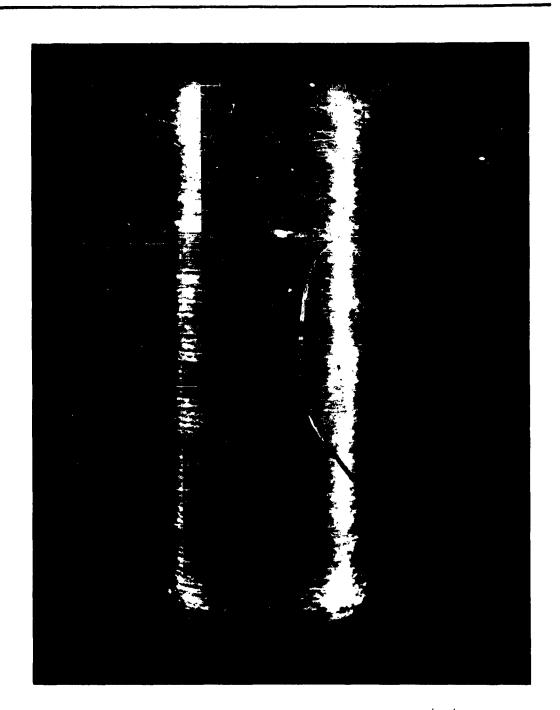


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of material, there was concern that the package may have been damaged by applying tension to it, and that this would affect the results on samples made from the underlying material. Results indicate no improvement on the second test where tension was applied to the strand. Therefore, applying tension to the package has not harmed the underlying material. Test values reported in Table 12 were too few to make a conclusive analysis, but the results obtained so far indicate that applying tension at a way-wound package of fresh material may not be harmful. However, this practice is not recommended, since damage might occur if a very soft package is used or if a package is used where the cohesive action of the resin would put severe unwinding loads on the strand, possibly causing degradation of the roving.

During performance of this work, observation was made of the effect on the package of applying tension at the package. When tension was applied to the package, there was no visible effect on the way-wind spool. However, the parallel-wound spool was damaged by the roving "digging" into the spool (Fig. 33). This occurred when the roving was fresh, and the package was relatively soft. After it was aged a few days at room temperature, the roving hardened sufficiently so the package was firm and not subject to indentation when tension was applied at the spool. Samples for the data presented in Table 12 were fabricated, using this spool after aging. No further damage occurred on the parallel-wound package. This experiment indicates one of the possible disadvantages of a parallel-wound package.

Another potential difficulty of a parallel-wound spool is a tendency for the package to elongate at the ends as it is wound. This condition would tend to exist only if the material were soft and sufficient tension were applied during windup. Under these conditions, flanges would need to be placed at the ends to support the package.



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Figure 33. Parallel-Wound Package of Preimpregnated Roving Where Tension Had Been Applied at the Package

TABLE 12

# EFFECT ON TEST CYLINDER OF APPLYING TENSION TO ROVING PACKAGE AND TO STRAND

Ultimate Hoop Tensile Stress, psi					
	Winding Tension				
Package Description	12 Pounds on Package	19 Pounds on Package	19 Pounds on Strand	12 Pounds on Strand	
Parallel" Wound	195,000 165,000	213,000 234,000			
75 Degrees Way-Wound	193,000 193,000	226,000 234,000	234,000 240,000		
75 Degrees Way-Wound (Repeat Test)	204,000 232,000	233 .000 242 ,000	230,000 242,000	230,000 213,000 220,000	

Certain disadvantages of the way-wind package have been noted. During high-speed winding, the strand traverses rapidly across the face of the spool and tends to abrade itself and the underlying material. No study of this condition was made. Roving feed rates used throughout the program were no higher than 100 ft/min.

Because of the helical pattern of windup of the package, wide spots in the roving band are created where the band reverses direction (turnaround point) on the spool. When tension is applied to the strand, the wide spot assumes its normal width condition where the roving is soft and pliable (but not if the roving has become hardened). This condition is shown in Fig. 7.

No conclusive evidence has been generated in this study to make a firm recommendation as to the design of the roving package. It is necessary to have more experience with each type before a decision as to which is best can evolve. However, there is sufficient evidence from the study above to indicate that the best winding procedure is to apply tension on the strand, and not at the spool. It also appears that a package with a high way-wound angle (greater than the 75-degree-angle package tested), but one that is not quite parallel, might be the best solution. This type of package would eliminate the digging into the spool on the parallel package and would reduce the abrading and widening effect on the 75-degree way-wind package.

#### Winding Machine Components

During this study, investigations were made to determine the effects of using pulleys of varying numbers, sizes and combinations, the suitability



of ceramic guide bushings for use with preimpregnated roving, the type of equipment suitable for preheating the roving, and the relative merits of various tensioning devices.

Guide Bushings and Pulley Systems. The nature of preimpregnated roving is such that special consideration must be given to the design of certain components of the winding machinery. Those considered here are the roving guide devices. It is especially desirable on numerically controlled winding equipment that the roving be guided from a fixed point on the delivery arm just before wrapping on the mandrel. Pulley wheels that pivot to line up with the angle of laydown of the roving are not considered entirely satisfactory. Consequently, consideration was made of a guide bushing of the type used as a thread guide in the textile industry. Comparative tests were made of a guide bushing made of titanium dioxide and of a steel roller pulley. The tendency of the roving to be degraded when passing over or through the device was measured by comparing hydrostatic burst test data of 3-inch-diameter test cylinders made, using first the metal pulley and then the ceramic guide bushing on the winding machine. The burst test data, converted to hoop tensile strength, are presented in Table 13. Although the results indicate slightly lower values when using the ceramic bushing, the difference is considered to be within experimental error. Use of a titanium dioxide guide bushing thus was considered satisfactory without fear of degradation of the preimpregnated roving.

However, in this case, the strand ran nearly stright through the ceramic guide bushing. Later experiments used similar guides mounted on the large, numerically controlled machine. The strand was bent at a sharp angle

TABLE 13

TEST RESULTS OF CYLINDERS WOUND USING CERAMIC BUSHING AND STEEL PULLEY ROVING GUIDES

Sample No.	Follower	Hoop Tensile Strength, psi		
1	Ceramic Guide Bushing	252,000		
2	Ceramic Guide Bushing	246,000		
3	Ceramic Guide Bushing	237,000		
4	Ceramic Guide Bushing	251,000 Average 244,100		
5	Steel Pulley	258,000		
6	Steel Pulley	272,000		
7	Steel Pulley	240,000		
		Average 256,666		

Difference: 256,666 -244,100

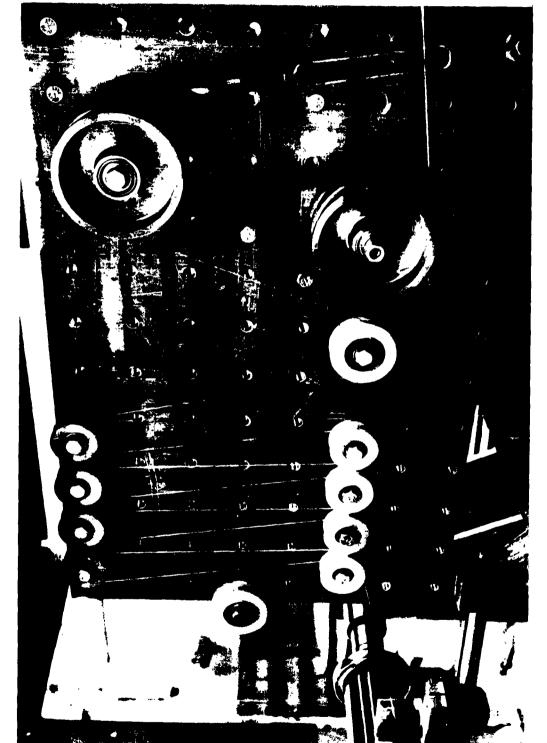
12,566

 $\frac{12,566 \times 100}{256,666} = 4.89$  percent

around the edge of a ceramic bushing during the winding operation. At the point of contact, excessive strand friction was noted and caused both filament abrasion and erratic variations in winding tension. Further evaluation of ceramic guide bushings was terminated. Use of these bushings is not recommended under these conditions.

Also of considerable interest in the design of components for winding machinery is the number and arrangement of guide pulleys that may be used without causing damage to the preimpregnated roving. To evaluate this problem, an experimental mounting plate was used to set up the various multiple pulley and brake combinations shown in Fig. 32, 34, and 35. In using the various setups, 3-inch-diameter cylinders were wound and tested hydrostatically. The test results, converted to hoop tensile stress, for each system tested are presented in Table 14. The spread of test results is within normal experimental variation\* for this type of testing, except for specimen PT-9 which is considerably lower than the others in the series and tends to unduly influence the average. However, even if this is taken into consideration, it would appear that there is a slight amount of deterioration occurring with the 10-pulley, 180-degree contact installation, compared to the 2-pulley setup. This is mitigated slightly by reducing the contact surface to 90 degrees. Another consideration negatively influencing the use of many pulleys is the possibility of tacky strands of material tending to wind around the pulley and to break. Consequently, it is recommended that the number of guide pulleys used in winding machinery be kept at a minimum for most rcliable operation.

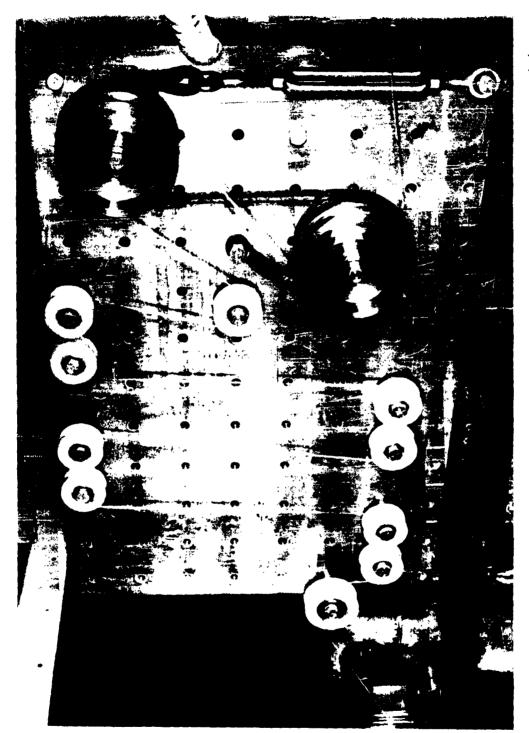
<sup>\*</sup>This statement is strengthened by the scatter of data where the low values of one set overlap the high values of the other.



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gure 35. Experimental Pulley Setup

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Sample No.	Pulley System Arrangements	Hoop Tensile Stress, 3-inch Dia Test Cylinders, psi
PT-4. PT-5	Feed system of 2 brakes, and 2 guide pulleys. Winding tension 18-20 lb	256,000 253,000
PT-6	on single 20-end strand (Fig. 10)	250,000
PT-10	(	251,000
PT-11		251,000
PT-12		240,000
PT-20		235,000
PT-21		246,000
PT-22		228,000
		Average 246,000
PT-1	Feed system of 2 brakes, 2 guide pulleys	234,000
PT-2	plus 8 guide pulleys added to system	242,000
PT-7	with 180-deg contact with roving strand	250,000
PT-8	(Fig. 11)	247,000
PT-9		<u>206,000</u>
		Average 236,000
PT-13	Feed system of 2 brakes, 2 guide pulleys	252,000
PT-14	plus 8 guide pulleys added to system	244,000
PT-15	with 90-deg contact with roving strand	244,000
PT-16	(Fig. 12)	250,000
PT-17		246,000
PT-18		233,000
PT-19		249,000
		Average 245,000

Note: All pulleys used were 1.5- and 2.0-in.-dia. nylon sleeves over ball bearings

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Materials for Pulleys and Guide Devices. Guide bushings and pulley spools made from Teflon were used to handle strands of preimpregnated roving in winding. Abrasion from the glass reinforcement in the strand rapidly cut into this relatively soft plastic.

A sleeve for a guide pulley was made from Kel-F. As in the case of Teflon, the glass reinforcement in the strand cut into the plastic surface of the pulley. Kel-F resisted abrasion better than the Teflon pulleys previously tested.

Nylon pulleys were found to be more resistant to abrasion than either Teflon or Kel-F. The Nylon rollers were found to be the most satisfactory of the plastic materials tested. The tendency of the roving to adhere to the Nylon was not significantly different from that found for Teflon or Kel-F.

Steel pulleys and guide rollers were used in connection with winding operations. The glass strand did not cause apparent damage to these surfaces. However, metal surfaces, in general, showed a greater tendency to pick up tacky resin than the plastic rollers evaluated.

Preheating Equipment. Preheating equipment was produced for use with the aging study. This equipment heated the roving just before placement on the mandrel by passing it through a chamber heated by a hot air flow (Fig. 3). The equipment was satisfactory, but the study indicated that preheating of the roving is not required. No further effort was expended.

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Tensioning Devices. Simple mechanical brakes were proved adequate where strand feed velocity was kept nearly constant. A mechanical device allowing dynamic friction laoding to be relatively stable over a wide range of strand feed velocity was tested and was found to be inadequate for low-end-count strands. The device is probably suitable for strands having a high end count.

As a result of problems encountered in using hysteresis brakes (reported by other companies under Navy contract), Rocketdyne did not believe this tensioning method required further study. A magnetic clutch was assembled for study as a tensioning and slack-takeup device. Evaluation could not be completed for inclusion in this report as it became apparent that a full investigation of tensioning devices must consider the entire delivery system and the winding procedure used. To define adequate tensioning procedures for any equipment exceeded the limits of this program.

#### Optimum Tensioning Technique

It was determined that, for small test cylinders, the tension should be applied to the strand rather than to the spool, regardless of package. Since the effects of strand abrasion are believed less for the small test cylinders than for large components, it is recommended that tension be applied primarily to the strand during winding.

#### Controlled Strand Tension

This study is related to the tensioning devices and complete delivery system used and was determined to be beyond the scope of this program. Considerably more effort regarding this subject is highly desirable.

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## Methods for Winding with Multiple Strands

Equipment for evaluating multiple-strand winding was constructed. One case was made with two strands of roving. Additional effort is required to evaluate the use of a larger number of strands in multiple-strand winding.

## Accurate Placement of Strands During Winding Operations

In experiments described in the next section, and in observations of strand placement, the NTC equipment produced a more precise pattern than did the mechanically controlled equipment; however, additional units would need to be made and tested on both types of equipment before a definite conclusion could be reached regarding the absolute influence of the precision strand placement.

In addition to winding cases to determine the relative precision of the NTC and the mechanically controlled machine, windings were made on test patterns for both types of equipment. This study was performed by wrapping a mercerized cotton thread around a mandrel and measuring the spacing between strands. Examples of such patterns are shown in Fig. 1, 28, and 29. These measurements have indicated that the NTC machine is capable of winding a pattern such as Fig. 1 shows to a tolerance on spacing of ± 0.01 inch. A tolerance of strand spacing as wound on the mechanically controlled machine was found to be approximately ± 0.06 inch.

#### CASE WINDING STUDIES

Under this portion of the program, six 18-inch-diameter x 24-inch-long cases were fabricated. Five of these cases were fabricated with the numerically tape-controlled winding machine. One case was fabricated on mechanically controlled winding equipment. For purposes of comparison of test data, the test results of a case previously made and tested under another program are included in the following discussion. This seventh case is identified as ABL 10.

The objectives of the program, the implementation of these objectives, the fabrication procedures used, the test methods followed, and a discussion of the test results are presented.

#### OBJECTIVES AND TEST PROGRAM

The basic objectives of the case winding studies were:

- 1. To compare the effects of the precise winding patterns performed by the numerically controlled winding machine with those produced with mechanically controlled equipment.
- 2. To study the effect of voids on the laminate stress level.
- 3. To compare the quality and strength of cases made with wideband roving with cases made with standard width roving.
- 4. To compare the use of wide-band roving made up with a number of strands laid down at once with the use of a single strand.

To implement these objectives, one case was fabricated using wide-band material, and the same mechanically controlled equipment used to fabricate the ABL 10 vessel. The ABL 10 vessel was fabricated with standard width roving; thus, a comparison was to be made of wide-band vs standard width band, as fabricated on mechanically controlled equipment. The vessel, fabricated under this program, was designated S/N 1.

To compare the quality and strength of cases made on a numerically tape—controlled machine to the cases made on the mechanically controlled equipment, two vessels were fabricated on the NTC machine; one of standard width band material and one of the wide-band material. These cases were designated as S/N 4 and S/N 5, respectively. To get a direct comparison between the cases made on the NTC machine with cases made on mechanically controlled equipment, the winding patterns and spacings selected were essentially identical on both types of equipment.

To study the effects of voids in a case, two vessels, S/N 2 and S/N 3, were fabricated on the NTC machine. The same type of roving was used for each vessel; however, the spacing and number of layers were different. With case S/N 2, spacing for the longitudinal layers was selected so that no voids resulting from narrow bands or band width variation could occur. Spacing of the circumferential winding was such that a 50 percent overlap of the band occurred. The circumferential windings were purposely applied to assure burst in the dome section of the tank for both S/N 2 and S/N 3. With the S/N 3 vessel, the longitudinal windings were applied in two layers with the band spacing such that it was exactly doubled that used for S/N 2. This spacing was greater than the average band width to ensure a gap between the strands as applied on the mandrel. The circumferential windings for S/N 3 and S/N 2 were equal in number, and weights of the cases were designed to be identical.

To compare the effect of winding with more than one strand to obtain a wide band, case S/N 6 was fabricated with two 20-end strands of roving, and at spacings double that of each layer applied in case S/N 2.

It was planned that all cases should be tested by hydrostatic burst without strain gages.

#### Fabrication

All of the cases were nominally 18 inches diameter by 24 inches long. The configuration conformed to the Allegheny Ballistic Laboratory design ABL 6400A. All cases were fabricated on mandrels of water-soluble plaster which was subsequently washed out of the cured case. The cases were constructed of E-HTS fiberglass roving preimpregnated with E787 resin system. In all cases, except S/N 6, a single 20-end strand was used. The width of the strand used for each case is shown in Table 15. Case S/N 6 was constructed using two 20-end strands.

Cases were constructed using a combination of longitudinal and circumferential windings (Fig. 36). The longitudinal fibers were placed at a 7-degree helical angle such that the strand progressed from the side of the metal boss at one end to the opposite side of the metal boss at the other end. This is sometimes called polar wrapping. The circumferential wrappings were extended slightly beyond the 13-1/4 inch length of the cylindrical portion of the case. For case S/N 1 through S/N 6, the longitudinal windings were sandwiched between the inner and outer layers of circumferential windings. The ABL No. 10 case (made and tested previously during a company-sponsored program) was made with all circumferential



DATA FOR 18-INCH-DIAMETER BY

TABLE 15

	ABL 10	s/N l	s,
Purpose of Test Equipment Used	Qualification, Mechanical	Wide Band, Mechanical	No Vo
Material			
Roving	One Strand 20-End E-HTS	Cne Strand 20-End E-HTS	0ne 20-E
Resin System	E-787	E-787	E.
Roving Bandwidth, nominal inch	0.085	0.125	0
Strand Spacing, inch			
Circumferential Longitudinal	0.045 0.074	0.045 0.074	0
Weight of Resin and Reinforcement, pound	4.66	4.36	7
Inside Diameter, inch	17.939	17.877	17
Wall Thickness, inch Total Longitudinal Plies Only	0.046 0.019	0.044 0.015	0.
Maximum Hydrostatic Pressure, psi	750	540	
Ultimate Tensile Stress, Laminate, psi Composite Unidirectional Laminate Longitudinal Plies Hoop Plies	146,000 178,000 249,000	110,000 162,000 167,000	176
Ultimate Tensile Stress, Fiberglass Only, psi Longitudinal Plies Hoop Plies	253,000 354,000	249,000 257,000	269
Resin Content, Percent by Weight	16.6	20.6	19
Burst Failure Location	Transition	Cylinder	Tran
Orientation of Fibers Where Failure Occurred	Longitudinal	Circumferential	Long

<sup>\*</sup> Total bandwidth of the two strands, as applied to the mandrel.

<sup>\*\*</sup> These cases designed to burst in dome sections and circumferential windings w therefore, composite wall and hoop plies stress is no true indication of perf

TABLE 15

DATA FOR 18-INCH-DIAMETER BY 24-INCH-LONG CASES

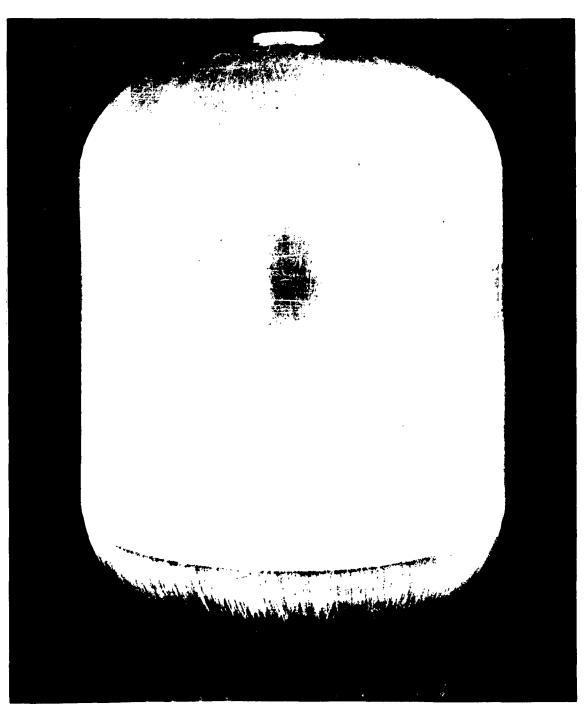
	S/N 1	S/N 2	s/n 3	S/N 4	s/n 5	s/n 6
on ,	Wide Band, Mechanical	No Voids, NTC	Voids, NTC	Narrow Band, NTC	Wide Band, NTC	Multiple Strand, NTC
s	One Strand 20-End E-HTS	Two Strands 20-End E-HTS				
	E-787	E-787	E-787	E-787	E-787	E-787
	0.125	0.090	0.080	0.085	0.125	0.145*
						<b>.</b> ".
	0.045	0.044	0.044	0.044	0.044	0.088
	0.074	0.055	0.110	0.074	0.074	0.110
	4.36	7.80	7.41	4.61	4.55	7.01
	17.877	17.875	17.854	17.826	17.824	17.876
	0.044	0.085	0.082	0.046	0.047	0.077
	0.015	0.085	0.025	0.017	0.047	0.077
	540	980	1025	650	920	1020
	-	·			,	
	110,000	**	**	126,000	174,000	**
	162,000	176,000	184,000	172,000	229,000	199,000
	167,000	**	**	200,000	285,000	**
	249,000	269,000	282,000	258,000	352,000	292,000
	257,000	**	**	300,000	435,000	**
	20.6	19.8	20.0	19.0	20.2	18.1
	Cylinder	Transition	Transition	Transition	End Dome	End Dome
1	Circumferential	Longitudinal	Longitudinal	Longitudinal	Longitudinal	Longitudinal



o the mandrel.

nd circumferential windings were thicker than required;

is no true indication of performance for these cases.



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Figure 36. Case S/N 4 Before Test

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windings placed over the longitudinal windings. During winding of the cases, the tension on the 20-end strand was adjusted to 16 to 18 pounds.

The wide-band roving was used in cases S/N 1 and 5. It was anticipated that a wide roving band, which is thinner, would nest and consolidate better than a thicker, narrower band. The material used was 20-end roving which had been processed by the supplier into a nominal 0.125-inch band. The uniformity of the band width was very poor, ranging from 0.080 to 0.150 inch. Where the band width was very wide, there was a tendency to separate into two bands. This occurred especially where the roving wound around the 75-degree way-wind package. The band also had a tendency to curl into a more narrow strand after leaving the last delivery pulley and before contacting the mandrel. Even though these deficiencies in the wide-band roving did exist, the nominal band width was still considerably wider than obtained with the standard material, and definite advantages over the standard band width material may be obtained with its use.

Since cases S/N 2 and 3 were designed to determine the effect of voids in the case, both cases were made with slightly more material in the hoop direction than was necessary for balanced construction to ensure failure in the end dome. S/N 2 was made with a combination of roving bandwidth and spacing to ensure overlap of the strand in the longitudinal winding. Case S/N 3 was made with a strand placement of 0.110 inch which was slightly greater than the 0.080-inch nominal bandwidth of the roving to ensure gaps and, consequently, voids in the laminate. The mechanics of fabrication required that the cases be made slightly heavier than the other cases in the series. The total weight of material in each case, S/N 2 and 3, was practically the same. During fabrication of case S/N 2 some difficulty was experienced in winding of the

longitudinal layers which required stopping and starting of the equipment Excess windings applied during restart operations had to be removed each time this occurred, and the tension of the roving was somewhat changed for these particular areas in the case. During removal of the excess windings, some of the longitudinal fibers were inadvertently cut in the cylindrical portion; this also may have had an effect on the performance of the case.

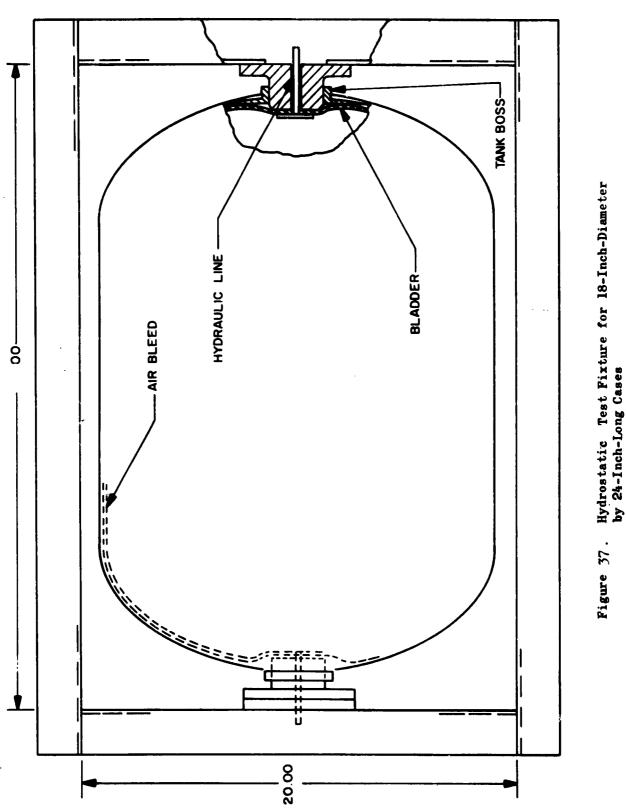
Difficulties similar to these which occurred on case S/N 2, described above, also occurred on case S/N 4; however, no fibers were cut on this case. Other defects also occurred in some of the cases. In two of the cases, small flat spots about the size of a quarter were caused by localized collapse of weak spots in the mandrel. In another a knot in the roving was wound into the structure. All of the known defects were carefully catalogued and their locations marked on the external surface of each cured case prior to hydrostatic testing. In no instance did failure occur at one of these defects, nor did the failed area include a section containing a known defect.

After fabrication, the cases were cured in a circulating hot air oven. The cure cycle consisted of 1 hour at 150 F, 1/2 hour at 250 F, 1 hour at 300 F and 3 hours at 350 F. Case S/N 1 was inadvertently submitted to a cure cycle where exposure to each temperature was approximately twice as long.

#### Testing

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Prior to hydrostatic testing, a rubber bladder was inserted in each case. Pressurization was performed with the case mounted horizontally in a fixture (Fig. 37). The pressure was raised at a steady rate to 550 psi in approximately 2 minutes. After a 1-minute dwell at this pressure, it was again increased at the same rate until failure.



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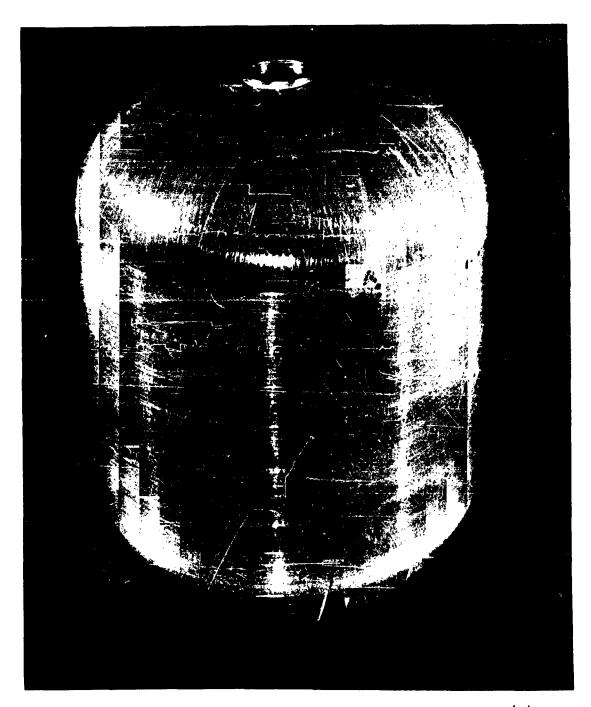
This occurred in the end domes by failure of the longitudinal fibers. Initial failure usually occurred at the transition point (illustrated in Fig. 38, 39 and 40) In cases S/N 5 and S/N 6, the failure appeared to initiate in the curvature of the end dome rather than directly at the point of transition (shown in Fig. 41 and 42). Although it appears that S/N 5 failed by blowing out the end boss, close examination indicates that the initial failure occurred in the longitudinal fibers in the curvature about 1 to 2 inches from the transition point. More than half of the fibers adjacent to the end boss remained intact.

The mode of failure for Case S/N 6' (Fig. 42) was similar to S/N 5. The construction of S/N 6 was similar to S/N 2.

Before and during testing it was noted that a considerable amount of crazing of the resin occurred. This was visible in the end domes only. The visible pattern of crazing was always parallel to the direction of the fiber. A diamond-shaped pattern occurred when crazing in both layers of the longitudinal wrap occurred and was visible through the surface resin. Crazing was most evident in S/N 2 and 3, probably because the case walls were thicker. Figure 43 illustrates the crazing evident before testing in case S/N 2. Crazing of this type had not occurred in cases previously made and tested at Rocketdyne

A number of days elapsed between the removal of the mandrel from case S/N 3 and the testing of this case. Crazing was evidenced as soon as the mandrel was removed; it grew progressively worse during the storage period between mandrel removal and case testing.

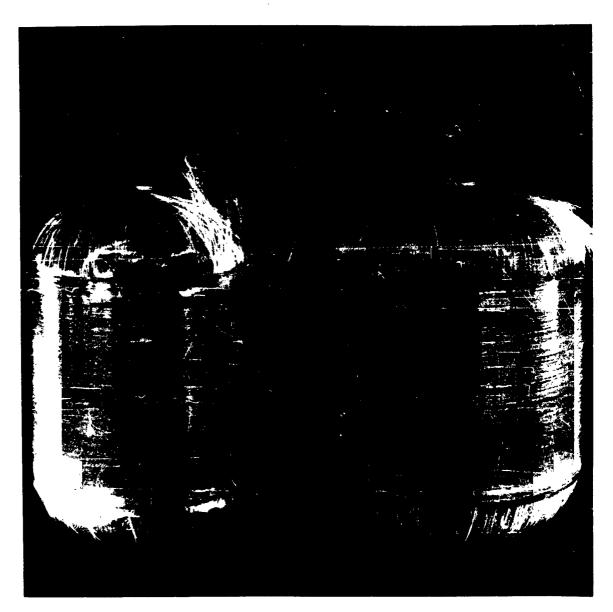
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Figure 38. Case S/N 4 After Burst Test

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Figure 39. Cases S/N 2 and S/N 3 After Burst Test

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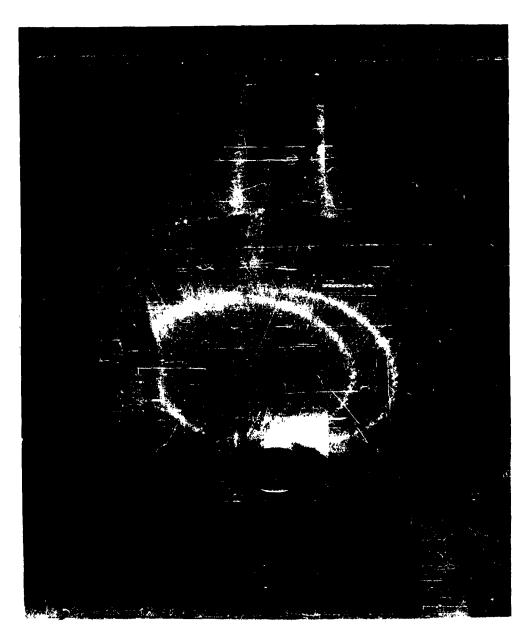


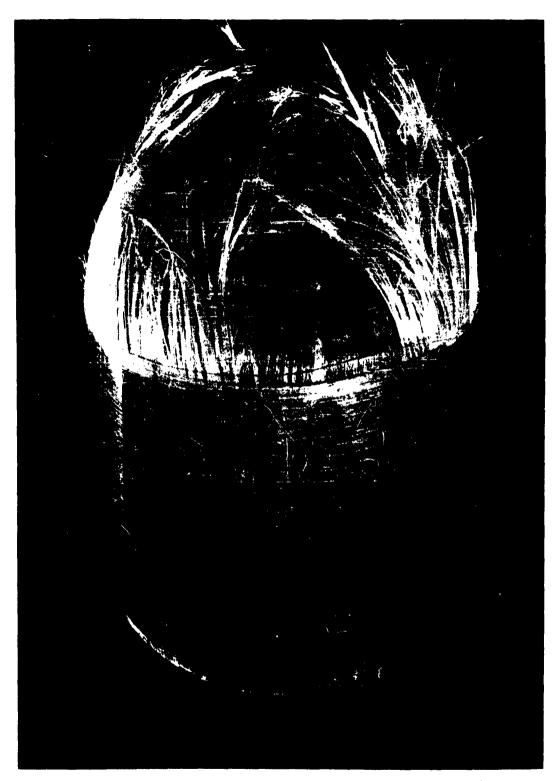
Figure 40. Case ABL 10 After Burst Test

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Figure 41. Case S/N 5 After Burst Test



X6920-7/5/62-1

Figure 42. Case S/N 6 After Burst Test



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Figure 43. Case S/N 2 Before Burst, Backlighted to Show Casing

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The exact causes for the crazing are not readily explained. Tension effects, resin shrinkage, and variations in coefficients of thermal expansion are probable factors contributing to the crazing observed. Considerably more effort would be required to determine either the causes or the effects of the crazing observed for these cases.

On examination of the cured laminate, resin-filled gaps between adjacent strands were evident, although these gaps did not appear to be quite so great as originally intended.

#### DISCUSSION OF TEST RESULTS

The data summarizing test results of the six cases made during this program, and the ABL 10 vessel, are presented in Table 15. Note that cases S/N 2, 3, and 6 were designed to assure burst in the dome section and, therefore, the results expressed are given in terms of the stress in the longitudinal direction only (i.e., stress was based on the thickness of the layer(s) containing the longitudinal filaments). Stress values presented are based on the laminate consisting of the combination of fiberglass and resin. This method of presentation includes the wall thickness contribution of the resin, and, therefore, is an indication of the strength-to-weight value. In addition, "glass stress" values are presented. In determining this value, the resin is assumed to make no contribution to the undirectional tensile stress and therefore should be discounted from the cross-sectional area of the material under stress. The value of this presentation is that the clouding effect of the resin content is removed from the analysis when making comparisons among a variety of test samples. In the following analysis, it should be noted, cases S/N 1 through 6 have nearly the same

resin content and can therefore be compared on the basis of either method of presentation of stress values. Case ABL 10 has a significantly lower resin content. Therefore, comparisons with the other cases should be based on glass stress values.

# Use of Wide Band Roving

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The use of wide-band material can be compared to narrow-band by comparing cases S/N 1 and 5 to ABL 10 and S/N 4. However, S/N 1 was inadvertently subjected to an incorrect cure cycle. It is believed that this cure schedule may have an adverse effect on the mandrel and, possibly, the case material, and thus upon the results obtained for the wall stress of this vessel. The results obtained on case S/N 1 are probably of little value and should not be included in the analysis. Consequently, S/N 5 remains as the only valid sample made with wide-band roving. Since the ABL 10 case was made on the mechanically controlled machine, the only valid comparison remaining is between S/N 4 and 5.

Case S/N 5, made with wide-band roving, performed significantly better than S/N 4. The stress which developed in the longitudinal plies where failure occurred was one-third better in S/N 5 than in S/N 4. Some difficulties were experienced with the functioning of the NTC machine during fabrication of S/N 4, requiring stopping and starting of the machine. On the other hand, the wide-band preimpregnated roving material was not uniform in width, varying from approximately 0.065 to 0.150 inches. It is difficult, then, to state without qualification that the wide band made a significant improvement in performance. Indications are that some definite benefit was derived from its use, but other test samples would be required to make a more firm conclusion.

## Effect of Voids on Stress Level

Cases S/N 2 and 3 were designed to determine the effects of voids and gaps in the case. As described earlier, the longitudinal windings in S/N 3 were spaced far enough apart to create gaps between the strands. It was intended that this would result in voids, or air pockets in the laminate. These voids, if present, would be visible because of the transparent nature of the structure. However, the resin flowed during cure and filled in the gaps to the extent that no significant amount of voids was visible. A comparison of the performance of S/N 2 and 3 shows no significant difference. The hydrostatic burst pressures were 980 and 1025 psi, respectively. The lower value for S/N 2 may have been due to the difficulties experienced during fabrication.

The calculated wall stress values, based on the logitudinal laminate thickness where failure occurred, were only 4 to 5 percent apart. It would seem that, based on these data, there is no significant effect on the case when there are gaps between the windings which are filled with resin.

No conclusions can be made regarding voids (air inclusions) in the laminate from these preliminary data since no voids were evident. These data indicate that, for a pressure vessel, gapping between the strands can be tolerated and may even be preferred to an irregular overlapping of strands. However, there is insufficient evidence, because too few samples were available to substantiate these conclusions. Further, it is often important with filament-wound vessels that the wound structure be stressed in compression or bending, as well as in tension. Although the effect of gaps may be insignificant with reference to tensile loads, the same degree of gapping in the laminate may quite possibly be quite detrimental

to performance with reference to either compressive or bending loads. On this program the controlled gap was approximately 35 percent of the band width during the winding, and the evidence obtained from the cured laminate indicated that an even lower percentage of gap existed in the cured structure. Considerably more effort should be expended, for the reasons listed, to define the allowable limits for gaps between strands in filament-wound structures. These additional studies should include not only hydrostatic loading of vessels but compressive and bending loading of vessels.

## Multiple Strands

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The use of multiple strands of preimpregnated roving combined into a single wide band was incorporated in the fabrication of case S/N 6. This case was designed to apply the same amount of roving per layer, and the same number of layers, which was used in case S/N 2. Two strands of 20-end, standard width band, preimpregnated roving were used during the winding. These strands were overlapped to form a band 0.145 inch wide. To obtain the same amount of roving per layer as used in case S/N 2, the strand spacing was doubled that for S/N 2. No difficulties were encountered during the winding of this case. No strand breakage or machine difficulties of any sort were encountered. In comparing the performance of S/N 6 to S/N 2 the difficulties experienced during fabrication of S/N 2 should be considered. The fact that a slightly greater stress level based on the thickness of longitudinally reinforced layers was obtained for S/N 6 than was obtained for S/N 2 can be partially explained by this lack of defects in S/N 6 which were present in S/N 2.

Although the indications are that multiple strands can be used with results equally as good as with single strands of roving, insufficient data are available to substantiate this conclusion. More effort is required to completely define the limits for processing with multiple strands of preimpregnated roving on the numerically controlled equipment.

# NTC Machine vs Mechanically Controlled Equipment

ABL 10 case, fabricated as part of another program, was selected for comparison purposes as representative of the best case made on the mechanically controlled equipment. Case S/N 1, 4, and 5, fabricated on the NTC machine, were basically the same contruction as ABL 10. Comparisons involving S/N 1 and 5 must consider that wide-band roving was used. However, S/N 1 is considered an unsatisfactory sample because of the improper cure cycle used, as discussed above. Direct comparison of cases made on the mechanically controlled machine and the NTC equipment can only be made between ABL 10 and S/N 4 and 5.

Case S/N 4--constructed in the same way as ABL 10, including the use of narrow roving--performed equally as well as ABL 10, comparing the glass stress of the longitudinal plies where failure occurred with each. In the light of the difficulties encountered during fabrication of S/N 4 which were not experienced during fabrication of ABL 10, the performance of S/N 4 can be considered superior to ABL 10.

Case S/N 5--constituted in the same way as ABL 10, except for use of wide-band roving--performed considerably better than ABL 10. This case, made on the NTC machine, developed during test a unidirectional laminate

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stress of 229,000 psi for the longitudinal ply and 283,000 psi for the circumferential plies. These values are based on the observed thickness of each ply. The stress in the circumferential direction compares favorably with an average value of 270,000 psi obtained on cylindrical test specimens on other programs using the same material. Reference is also made to the glass stress of 435,000 psi in the hoop direction. This value approaches the ultimate monofilament strength of 500,000 psi for E-HTS glass. It seems possible that, since burst failure did not occur in the hoop direction, the developed stress was not the ultimate possible for the hoop fibers in this case. No difficulties were encountered during fabrication of case S/N 5. It is entirely conceivable that this case represents a performance level that can be readily duplicated on the NTC machine.

Cases S/N 2, 3 and 6, made on the NTC machine, were not constructed as ABL 10 was, and a direct comparison of performance theoretically can not be made. However, these cases failed during test in the same manner as ABL 10 failed. All three, evaluated on the basis of glass stress in the longitudinal plies, performed better than did ABL 10. It is now apparent that all the cases made on the NTC equipment developed a higher glass stress in the longitudinal plies where failure occurred than did ABL 10. Since ABL 10 represented the best made on the mechanically controlled equipment, it can be concluded that the NTC machine can readily produce a superior filament-wound product using preimpregnated roving.

## PROCESS SPECIFICATION

A process specification which details limits of storage, handling of the preimpregnated material, and the processing of the #787 preimpregnated roving into filament-wound components is presented in the Appendix. The limits presented in this specification are based on the work done under this program. To have more exact limits in this specification, and to allow this specification to call out limits on materials properties or processing procedures not covered under this specification, considerably more effort will be required.

It is possible that, with some efforts directed toward correlation of data obtained under this program and that obtained by other investigators, a more comprehensive specification could be written without an extensive testing program. Although some of these data are available to Rocketdyne, the tests required to correlate these data with the Rocketdyne data were beyond the scope of this program.

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#### RECOMMENDATIONS

As a result of the investigations performed in this program there is definite evidence that the use of preimpregnated roving with numerically controlled winding equipment results in parts with superior strength properties. Due to the limited nature of the program, however, many of the tests run could only be carried out on one part. The data obtained can therefore only indicate a trend rather than a positive number. In order to validate some of the conclusions and to arrive at absolute values for some of the parameters, it is recommended that further work be carried out in the following areas.

- 1. Comparison of numerically tape controlled and mechanically controlled winding equipment
- 2. Effects of gaps and voids
- 3. Wide-band vs narrow-band roving
- 4. Comparisons between strand tensile strength and stress levels in the finished part, and use of strand strength test for quality control of the roving.
- 5. Use of high-angle way-wind packages
- 6. Effects of resin migration and methods of control.

Due to the superior results obtained with numerically controlled equipment it is also recommended that work be initiated to compare various head shapes and winding patterns by making parts on the numerically controlled equipment so as to eliminate the factor of inaccurate winding from such a comparison.

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During this program and in the course of other work being performed at Rocketdyne, it was observed that the resin in preimpregnated roving would exhibit different flow characteristics. Variations in laminate wall thickness would result and also in the developed stress level. This was probably caused by variations in the degree of B-staging and influenced by the various cure schedules used. This flow characteristic appears to have a significant effect upon strength properties. The influence of flow and the factors that affect it are recommended for study since they could not be covered in the present program.

During fabrication of cases for this program it was necessary to splice roving into the windings where breaks occurred or restarts were necessary. It was thought these splices had a detrimental effect upon the performance. The need for splices will ever be present, and the effect of splices more significant as the performance demand on the material is increased. It is therefore recommended that the influence of splices on strength be studied and that effective splicing procedures be developed.

#### APPENDIX

DESIGN AND TEST PROCEDURE OF 3-INCH-DIAMETER BY 6.37-INCH-LONG CYLINDRICAL TEST SPECIMEN OF FILAMENT-WOUND CONSTRUCTION

#### **RACKGROUND**

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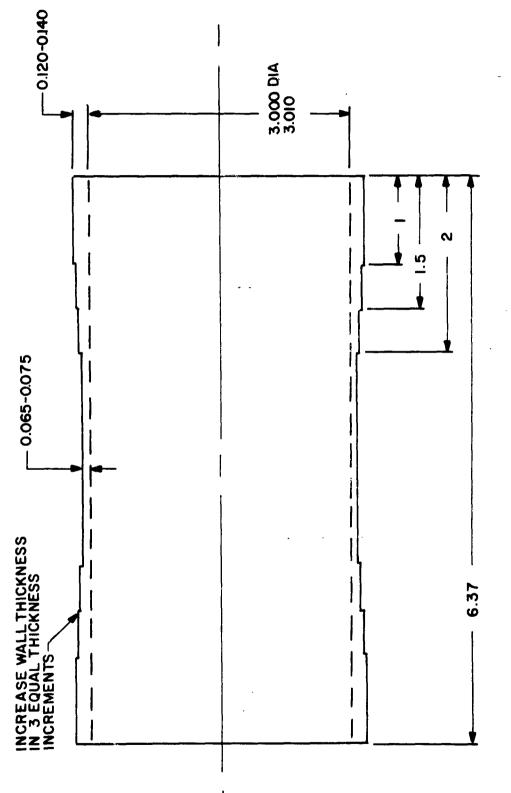
A 3-inch-diameter by 6.37-inch-long cylindrical sample has been used at Rocketdyne for measuring pure hoop tensile strength of filament winding materials for several years. Testing has been performed by applying hydrostatic pressure to the inside of the cylinder until burst failure. This test has been used for evaluating materials and process parameters.

#### SPECIMEN CONFIGURATION AND DESIGN

Dimensional details of the test cylinder are shown in Fig. 44 Construction is performed by circumferentially wrapping these materials on a metal mandrel:

Fiberglass roving (wet system or preimpregnated), inches thick 0.030
One ply 158430 fabric (dry or preimpregnated), inches thick 0.013 to 0.017
Fiberglass roving, inches thick 0.030

The fabric is wrapped over the first layer of roving in exactly 1 ply. The ends are brought together in a butt joint. The fabric is placed on the mandrel so that the warp fibers are parallel to the axis. If preimpregnated roving is being used, then the fabric must be impregnated with the same resinhardener system. If the wet system is being used, the dry fabric will absorb



Filament-Wound Cylindrical Test Specimen

Figure 44.

the excess resin in the roving. The additional thickness at each end of the cylinder (Fig. 44) consists of circumferential wrappings added to the basic structure. The buildup at the ends ensures failure in the central area of the cylinder and, also, reduces expansion in the area of the cup seals of the hydrostatic test fixture to ensure maintenance of the seal.

Note that the construction has been designed for determining pure hoop tensile stress of a circumferentially wound structure. The unidirectional fabric is included to prevent premature failure from bending loads. Construction of the fabric is:

#### Thread count:

Warp direction, per inch	42: 150's, 4 x 2	
Fill direction, per inch	30: 450's, 1 x 3	
Thickness, inches	0.0131	
Weight, oz/sq yd	15.18	
Breaking strength, dry fabric:		
Warp direction, lb/in. width	1200	
Fill direction, lb/in. width	65	

The fabric is manufactured by United Merchants Co.

#### STRESS CALCULATION

The longitudinal fibers (warp fibers of the 158430 fabric) are considered to make no contribution when hoop tensile stress is being calculated. Because of the very low strength of the fill fibers in comparison to the total strength of the roving, and because in the circumferential direction

the fill fibers are discontinuous at the butt joint of the fabric, the contribution of these fibers is considered insignificant. Consequently, the thickness of the layer of longitudinal fibers (0.013 to 0.017 inch) is subtracted from the total wall thickness to determine the net wall thickness. The fabric thickness is measured on the exposed material in the broken specimen. The hoop tensile stress is then calculated, as follows:

$$\sigma = \frac{\mathbf{p_r}}{\mathbf{t}}$$
, pai

where

P = hydrostatic pressure at burst, psi r = mean radius of specimen, inch = 1.5 + 1/2 total wall thickness t = net wall thickness, inch

The stress calculated is the unidirectional stress in the resin-glass laminate. This should not be confused with fiber stress which is based on the fiberglass filaments only.

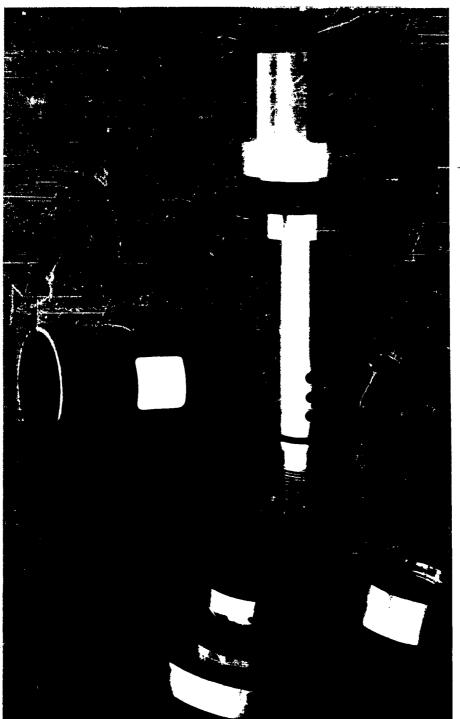
Measurement of the wall thickness is generally made with micrometers which have spherical-shaped anvils. Unless specified otherwise, the wall thickness value includes surface resin. Measurement is made adjacent to the point of failure, or, if made before testing, the thinnest wall section is determined. Four points around the circumference are measured. The lowest or next lowest value is used, dependent on the spread of thickness values. (Failure is assumed to occur at the thinnest wall, but a tolerance on measurement is assumed to exist.) Choice of the lowest or next lowest value requires a certain amount of judgment based upon knowledge of the construction of the sample and measurement techniques.

#### TEST PROCEDURE

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Prior to hydrostatic testing, a nonpermeable sleeve such as latex rubber is placed inside the speciment to prevent leakage of the pressurizing fluid. The specimen is placed on a fixture (Fig. 45) which traps the hydrostatic pressure between the cup seals. The fixture is designed so that no end loads are applied to the cylinder and no points of stress concentration are created. Pressurizing fluid (water at room temperature) is introduced into the specimen, creating a hydrostatic pressure. Care must be taken to expel all air from the cylinder. Buildup of this pressure is contained at a uniform rate of 300 psi/sec until failure (Fig. 46). Hydrostatic pressure is measured, using a suitable pressure gage or automatic recording equipment.

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Cylindrical Test Specimen and Hydrostatic Test Fixture Figure 45.

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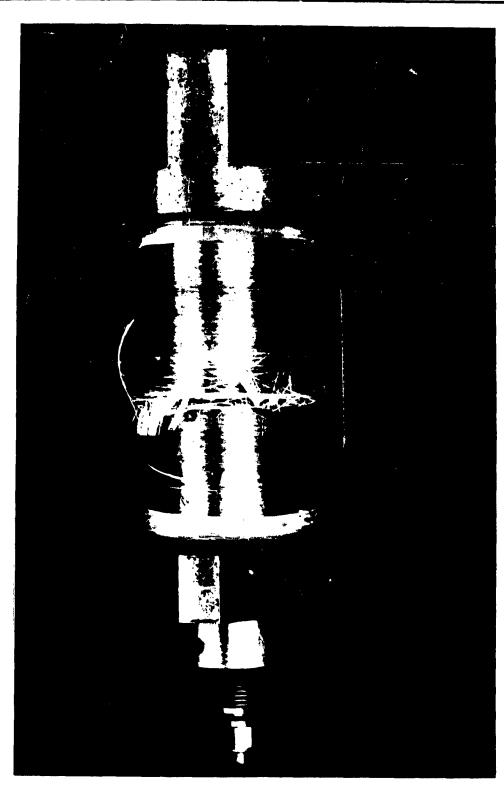


Figure 46. Burst-Tested 3-Inch-Diameter Test Cylinder

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#### INTERLAMINAR SHEAR TEST

The interlaminar shear test used in the preceding study was that described by Perry, et al, in the paper, "Status of the NOL Ring Test for Glass Roving Reinforced Plastics."\* A ring 0.250 inch wide is cut from a cylinder constructed similarly to that used for hoop tensile stress, except total wall thickness was 0.125 inch, and the outside diameter was sanded smooth. The speciman is cut from this ring in the circumferential direction and tested in bending over a short span to induce failure in horizontal shear (Fig. 47).

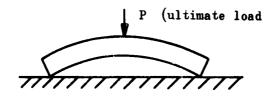


Figure 47. Interlaminar Shear Test

The support surface is very smooth and may be lubricated to allow lateral movement. The specimen has a chord length of 0.500 inch, a width b=0.250 inch, and a thickness t=0.125 inch. Shear stress is calculated by this formula:

$$\tau = 3/4 \frac{P}{bt}$$
, psi

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\*1961 SAMPE Filament Winding Symposium, Pasadena, California

#### GENERAL PROCESS SPECIFICATION FOR USE OF PREIMPREGNATED ROVING

#### 1. INTRODUCTION

#### 1.1 SCOPE

This specification describes the materials and processes used in the filament winding of high-strength, lightweight pressure vessels, rocket motor cases, etc., using continuous fiberglass filament preimpregnated with epoxy resin.

#### 1.2 CLASSIFICATION

Structures are of types requiring close dimensional tolerances, laminate uniformity, and high performance reliability.

#### 1.3 LIMITATIONS

Parameters and requirements herein are based on data developed for one particular epoxy resin-preimpregnated glass filament system and are not necessarily applicable to all systems.

#### 2. APPLICABLE MATERIALS AND SPECIFICATIONS

#### 2.1 MATERIALS, PRODUCTIVE:

#### Material

Fiberglass Roving, 20 end, E-801, Preimpregnated with Epoxy Resin E787 U. S. Polymeric Chemicals, Inc.

Santa Ana, California (Appendix, 151)

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## ROCKETDYNE A DIVISION OF NORTH AMERICAN AVIATION, INC.

Fiberglass Roving, 20 end, E-HTS, Preimpregnated with Epoxy Resin E787 U. S. Polymeric Chemicals, Inc.Santa Ana, California(Appendix, 151)

## 3. STORAGE OF MATERIALS

- 3.1 Spools of preimpregnated roving shall be kept in the original, or equivalent, sealed bag or envelope and protective container in such a manner as to prevent contamination or damage of the material.
- 3.2 Material shall be stored prior to use at the following temperatures for the period of time specified.

Temperature, maximum, F	Time, maximum
80 '	72 hours
40 .	6 weeks
0	12 weeks

Storage time shall be determined from the date of impregnation. Times at different temperatures shall not be accumulative. That is, for example, material that has been stored for 10 weeks at 0 F may not be subsequently stored for 6 weeks at 40 F without requalification. For determination of maximum allowable accumulative storage time, the following table of equivalents may be used:

24 hours at 80 F = 1 week at 40 F = 2 weeks at 0 F

- 3.3 After storage for the maximum period of time as determined in 3.2, material shall be tested for compliance with the requirements of the material specification for preimpregnated roving.
- 3.3.1 Requalified material will be subject to qualification testing at the end of 3 weeks when stored at 40 F, and 6 weeks when stored at 0 F.
- 3.3.2 Material meeting the test requirements must be used immediately.
- 3.3 Lots of material placed in storage shall be removed on a firstin, first-out basis.
- 3.4 Material that has been removed from cold storage shall remain in the sealed protective cover until reaching room temperature before being used.

## 4. MACHINE COMPONENTS

- 4.1 DELIVERY PULLEYS
- 4.1.1 Pulley wheels used to support the roving band between the package and mandrel shall be as large as possible, preferably no less than 1.5 inches diameter.
- 4.1.2 The number of pulleys in contact with the roving shall be as few as possible, prefcrably less than eight, and the area of contact that the roving makes with the pulleys shall be less than 180 degrees.

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- 4.1.3 Pulleys shall have a nonsticky contact surface and shall rotate freely. To meet these requirements, they shall be constructed of nylon or Teflon (or Teflon-coated metal) sleeves firmly attached to a ball bearing mounted firmly on a fixed metal stud or shaft.
- 4.1.4 The first delivery pulley contacted by the roving shall be located from the surface of the spool at a minimum distance equivalent to the length of the package. This measurement shall be made normal to the axis of spool. Guide rollers with axis mounted normal to the axis of the package shall be used between the spool and first pulley immediately adjacent to the pulley.

#### 4.2 TENSIONING DEVICES

- 4.2.1 Tension control devices shall be capable of maintaining a tension on the strand within a tolerance of  $\pm 5$  percent of the nominal setting at all rates of roving feed velocity.
- 4.2.2 The surface of brake rolls contacted by the roving shall be relatively nonslippery and not subject to corrosion that may harm or contaminate the roving.
- All pulley wheels, and other surfaces, contacted by the roving shall be kept free from transposed resin and any foreign materials that may contaminate or harm the roving strand. Pulley wheels, and other devices, contacted by the roving shall be replaced when the surface of contact becomes rough or distorted so as to possibly cause harm to the roving strand.

## 5. MANDRELS

- Mandrels may be constructed of materials such as plaster, water-soluble salts, high-temperature melting salts, all metal, or combinations of metal and other materials. They shall be dimensionally stable, strong enough to support the applied winding loads, and entirely satisfactory for the purpose intended.
- The mandrel surface shall be covered with a mold release that will allow easy separation of the mandrel from the filament-wound structure. In the case of mandrels made from plaster or other water-containing materials, the mandrel surface shall be covered with a nonpermeable sealant film prior to application of the mold release. Mold release and sealant materials shall have adequate resistance to cure temperature, and shall be entirely compatible with the materials in contact with them.

## 6. WRAPPING OPERATION

- 6.1 The mandrel shall be installed in the winding machine within the limits specified for axial alignment and concentricity during rotation. All other aspects of mandrel location and alignment shall be checked for compliance with detailed instructions.
- Preimpregnated roving strand(s) shall be threaded over the appropriate brake and delivery pulleys without twists. During the winding operation, twists shall be removed from the strand(s) when their existence results in the introduction of twisted strands into the filament-wound laminate.

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- 6.3 The last delivery pulley or guide contacted by the roving prior to wrapping on the mandrel shall be placed as close as practical to the mandrel, or at the distance required by the detailed instructions for the part being fabricated.
- Tension on the strand(s) shall be measured between the last pulley or guide contacted by the roving and the mandrel. Tension of the strand(s) shall be measured while the strand is moving at the velocity of the actual winding operation, using a Kidde tensionmeter or similar device.
- Tension shall be applied to the strand by a system of brake rolls over which the strand traverses. The total tension shall be 18 ± 2 pounds for each 20-end strand, unless specified otherwise. A maximum of 15 percent of the total tension shall be applied at the spool by resisting the rotation of the package. The total tension shall be monitored throughout the winding operation, or checked periodically with a device capable of making measurements on the moving strand.
- Before and periodically during winding, the turns per inch being applied by the machine shall be checked for compliance with detailed requirements.
- 6.7 Restarts in the midst of the winding operation necessitated by breaks in the strand, runout of a spool of material, or breakdown of the winding equipment shall be accomplished as follows:
  - a. The strand, while under tension, shall be cut close to the mandrel and the cut end firmly pressed against the

mandrel or previously applied wrappings. The cut shall be made in a location that will have the least effect upon the strength of the fabricated item. For example, longitudinal filements shall be cut in the cylindrical area of a pressure vessel or motor case.

- b. Restart shall be made by placing windings over two or three turns of the previously applied wrappings of the same layer. After progressing a minimum of three turns past the stopping point, the excess (overlapping) windings shall be carefully peeled off.
- c. Where multiple strands are used and a break occurs in one strand, the new strand may be attached to the unbroken strand. at the end of the broken strand so as to allow the new strand to be carried to the mandrel. The new strand shall be attached by pressing it to the unbroken strand over a contact length of 12 inches at a convenient location along the roving delivery system of the machine. Adhesion of the strands may be implemented by use of a roller device to obtain greater contact pressure and heat from a hot air gun. No more than one strand at a time may be so attached.

CAUTION: Roving shall not be heated above 125 F.

6.8 Winding will be performed at room temperature unless specified otherwise. Where it is desirable, or necessary, to preheat the roving, it shall be heated to a maximum temperature of 125 F. Heat shall be applied to the strand as close to the mandrel as possible.

- 6.9 Heat may be applied to localized areas on the wrapping during the winding operation in order to soften the roving and facilitate laydown as it is being wound around bosses, to increase tackiness in order to improve adhesion and reduce slippage where necessary, or for other requirements to facilitate winding.

  CAUTION: Roving shall not be heated above 125 F.
- 6.10 Visual inspection shall be maintained during the winding operation of each spool of preimpregnated roving. Use of the spool shall be terminated and the spool replaced by a new one when the presence of any of the following defects becomes excessive and threatens to degrade the quality of the part being fabricated.
  - a. Bandwidth outside of tolerance
  - b. Nonuniform impregnation, as indicated by dry roving
  - c. Fuzziness of the strand
  - d. Nests and knots in the strand
  - e. Twists in the strand
  - f. Folds parallel to the fibers (apt to occur in wide-band material).

#### 7. CURING PROCEDURE

7.1 Cure schedule shall be that specified for the material used.

Temperatures shall be measured by thermocouples attached to the coldest portion of the resin-glass structure. An autographic record of time-temperature relationship shall be made for each part.

- 7.2 External pressure applied to the filament-wound laminate by the use of a vacuum bag or shrink tape may be used only when required by the detailed specification for a specific part.
- 7.3 Parts shall not be cured in an oven previously used to cure materials that may contaminate the part.

#### 8. FINISHING OF PARTS

- 8.1 No sanding, machining, or blasting operations may be performed on plastic materials in areas of structures subjected to internal pressurization, except as noted on applicable drawings.
- 8.2 All finishing operations shall be performed according to applicable specifications or drawings only.

## 9. REPAIRS

- 9.1 No repairs may be made to primary structures after cure, except as approved by Material Review Board.
- 9.2 Defects in uncured laminates may be repaired by removing the defective material and reperforming the applicable winding operation.

  Removal of defective material shall be performed so as not to cause damage to the remaining material.
- 9.3 Repairs shall be performed only upon the approval of and under the supervision of the Quality Control Department.

10. QUALITY CONTROL

#### 10.1 INSPECTION

- 10.1.1 Parts shall be inspected to the dimensional requirements of the drawings.
- 10.1.2 Cured parts shall be inspected for defects that would affect the structural integrity of the part.
- 10.1.3 In-process inspection shall be performed according to the requirements of the detailed fabrication specification for specific parts and the requirements of this specification.
- Where necessary for closer control of the roving, and for a more complete history of the material used in the part, each package of material may be sampled and inspected. The tests required and the frequency of testing will be as specified by the detailed requirements of each part.

#### MATERIAL SPECIFICATION FOR GLASS ROVING, EPOXY RESIN IMPREGNATED, UNCURED

#### 1. SCOPE

This specification establishes the requirements for three types of continuous glass filament roving impregnated with a B-stage, heat-curing, epoxy resin system suitable for filament winding.

#### 1.1 CLASSIFICATION

## 1.1.1 Types

The impregnated roving shall be of the following types, as specified:

Type I General purpose

Type II High tensile strength

Type III Very high tensile strength

## 1.1.2 Classes

The impregnated roving shall be of the following class unless otherwise specified:

Class 20 20 end roving

## 2. APPLICABLE DOCUMENTS

None

## 3. REQUIREMENTS

## 3.1 MATERIALS

The finished preimpregnated roving shall consist exclusively of a suitably finished fiber glass roving, a heat-curing, B-Stage, epoxy resin system and a small amount of residual solvent. The materials used shall be uniform in quality, free of foreign materials, diluents and contaminants, and shall be entirely suited to the intended purpose.

#### 3.1.1 Roving

The roving used shall be:

Type I. Low alkali, lime-alumina, borosilicate glass known commercially as ECG 140's, with a suitable, epoxy-compatible size.

Type II. Low alkali, lime-alumina, borosilicate glass known commercially as ECG-140's, with a suitable, high-tensile-strength epoxy-compatible size which is applied at the bushing.

Type III. Low alkali, magnesium aluminum silicate glass of improved tensile strength, with a suitable, high-tensile-strength epoxy compatible size which is applied at the bushing.

The required number of ends, as specified by the class number, shall be gathered without twist. The end count shall be as specified  $\pm$  0 ends.

## 3.1.2 Resin System

The resin system used shall be an epoxy resin system suitable for preimpregnation and B staging. The curing system selected shall be capable of being cured and postcured at temperatures not in excess of 350 F for periods of time not in excess of 6 hours. High boiling, reactive or nonreactive diluents or extenders shall not be used.

## 3.1.3 Resin Content

Unless otherwise specified the nominal resin content shall be 20 percent.

## 3.2 PHYSICAL PROPERTIES

The preimpregnated roving shall meet the physical property requirements of Table 16 when tested by the methods indicated.

TABLE 16

PHYSICAL PROPERTIES OF PREIMPREGNATED ROVING

				Required Value	
Property	Units	Test Method Paragraph	Type I C1 20	Type II Cl 20	Type III Cl 20
Volatile Content	weight percent		3.0 maximum	3.0 maximum	3.0 maximum
Resin Content	weight percent		nominal ± 2.0	nominal ± 2.0	nominal ± 2.0
Glass Weight	grams/yard		0.621-0.673	0.621-0.673	0.604-0.654
Resin Flow	weight percent		8 minimum	8 minimum	8 minimam
Tackiness	inches		40 maximum	40 maximum	40 maximum
Band Width	inches	٠	0.075 minimum	0.075 minimum	0.075 minimum
Strand Tensile Strength	spunod		95 minimum	*	*
Hoop Tensile Strength	pai				
(1) Room Temperature			240,000 min	260,000 min	*
Interlaminar Shear Strength	psi				
(1) Room Temperature			6500 minimum	7500 minimum	*
(2) At 300 F			*	*	*
(3) At Room Temperature after 2 hour water boil			*	*	*
		1	7		

\* Values to be established.

#### 3.3 BANDWIDTH

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The roving shall meet the bandwidth requirement of Table 16 and shall be a single web of roving without splits or separations.

#### 3.4 KNOTTING AND SPLICING

There will be noknots or splices introduced during the resin impregnation process.

#### 3.5 WORKMANSHIP

Roving submitted for qualification or acceptance under this specification shall be of the highest quality of workmanship and shall be free of manufacturing defects which would adversely affect the performance of the finished roving. Visible indications of dry streaks or other evidences of poor impregnation shall be cause for rejection.

#### 3.6 SHELF LIFE

The preimpregnated roving shall meet all of the requirements of this specification following storage for 6 months when shipped and stored at temperatures not exceeding 0 F.

3.7 The roving shall be capable of being unwound easily and rapidly under a tension equivalent to  $4 \pm 0.4$  pounds per 20-end strand and shall show no tendency to dig into the ball or to leave loose ends or strands.

#### 3.8 QUALITY ASSURANCE

## 3.8.1 Qualification Tests

The qualification tests shall consist of all of the tests of this specification conducted on a sample which is entirely representative of the properties and packaging of the material to be offered for purchase to the requirements of this specification.

## 3.8.2 Material and Process Changes

Following approval of a supplier's qualification sample the supplier shall make no changes in the materials or processes used in the manufacture of the impregnated glass roving without prior approval of the purchaser. Material or process changes may require requalification of the material or other evidence of acceptability at the option of the purchaser.

#### 3.8.3 <u>Lot</u>

A lot of impregnated glass roving shall consist of not more than one lot of unimpregnated glass roving, impregnated in a single, continuous operation with a single batch of resin and offered for inspection at one time.

#### 3.8.4 Certification

Each lot of impregnated glass roving offered for inspection shall be accompanied by the manufacturer's certification that the glass,

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resin system, and processing used in the manufacture of the material being submitted are identical in every way to the glass, resin system, and processing used in the manufacture of the supplier's qualification sample.

## 3.8.5 Lot Acceptance Tests

Lot acceptance tests shall consist of any or all of the tests of this specification at the option of the purchaser. The frequency of the performance of any of the tests and any sampling plans shall be determined by the Rocketdyne Quality Control Department. Any defects which were not detectable during lot acceptance testing and which became apparent during the subsequent use of the material shall be cause for rejection of the unused portion of the ball, provided that such defects are cause for rejection under the requirements of this specification and are not a result of mishandling, improper storage, or expiration of shelf life.

## 4. METHODS OF TEST

#### 4.1 SAMPLING PROCEDURES

## 4.1.1 For Hoop Tensile and Shear Strength Tests

The sealed package shall be removed from the storage environment and shall be allowed to come to room-temperature condition. The package shall then be opened, and, after removing and discarding the first three way winds, the material shall be wound directly

from the sample roll onto the appropriate mandrel to prepare the test specimens. The sample roll shall then be resealed in the original container and returned to the storage environment.

## 4.1.2 Other Specimens

While at the storage temperature, the package shall be opened and, after removing and discarding the first three way winds, sufficient material for the tests to be run shall be taken from the roll. The samples shall be immediately sealed in moisture-proof bags, identified, and allowed to come to room-temperature conditions. The roll shall then be resealed in the original container. The samples shall be tested within 8 hours after being taken from the roll. If such sampling results in damage to the material, all sampling shall be conducted in accordance with paragraph 4.1.1.

#### 4.1.3 Storage Records

Records shall be maintained of the periods that any roll has been out of storage environment.

#### 4.2 TEST PROCEDURES

The following test procedures shall be used:

#### 4.2.1 Volatile Content

Three samples, each 36 inches ± 0.1 inch long, shall be weighed to the nearest milligram. The loosely coiled specimens shall be

dried for 15±1 minutes in a circulating air oven maintained at 225±10 F. Each of the specimens shall be removed from the oven, cooled in a dessicator and reweighed to the nearest milligram. Calculate the volatile content as follows:

Volatile Content, weight percent = 
$$\frac{W_0 - W_1}{W_0} \times 100$$

Where Wo = original weight in grams.

Wi = Weight of sample with volatile removed.

The average of the three values shall be reported.

## 4.2.2 Resin Content

The three samples used for volatile content shall be placed in previously ignited, cocled, and weighed porcelain evaporating dishes or crucible and shall be ignited for 60±10 minutes in a furnace maintained at 1150±50 F. The samples shall be cooled in a dessicator and reweighed to the nearest milligram. Calculate the resin content as follows:

Resin Content, weight percent = 
$$\frac{W_1 - W_2}{W_0} \times 100$$

Where Wo = original weight in grams

 $W_1$  = Weight of specimen with volatiles removed in grams

W<sub>2</sub> = Weight of specimen after ignition in grams

The average of the three values shall be reported.

4.2.3 Glass Weight per Yard

The weight of each of the specimens after ignition (W<sub>2</sub>) shall be recorded as the weight per yard. The average of the three values shall be reported.

4.2.4 Resin Flow

Resin flow shall be determined as follows:

- 4.2.4.1 Cut six lengths of roving, each 3 inches long. Weigh each specimen, consisting of two 3-inch lengths, to the nearest 0.001 gram.
- 4.2.4.2 With the lengths spaced approximately 1/2 inch apart, sandwich each specimen between two layers of 112-weave, volan-finished glass cloth or equivalent.
- 4.2.4.3 Press the sandwiched material on a flat plate maintained at 300±10 F with a 1500±15 grams metal weight preheated to 300±10 F for 2.0±0.1 minutes.
- 4.2.4.4 While still hot, remove the glass cloth containing the resin flow.

  Reweigh each specimen. Calculate resin flow as follows, and report the average value of tests on three specimens:

Resin flow, weight percent =  $\frac{W_3 - W_4}{W_3} \times 100$ 

Where W<sub>3</sub> = Weight of roving before cure in grams.

W<sub>4</sub> = Weight of roving with resin flow removed (after cure)
in grams.

#### 4.2.5 Tackiness

Two strands of roving shall be placed on the horizontal track of the tackiness test fixture shown in Fig. 20. The roving strand and the fixture shall be at 75 to 80 F at the time of test. The roving strands shall be placed 0.375 inches from the apex of the "V" and shall be held under a tension equivalent to 4±0.4 pounds per 20-end strand. A polished steel ball 0.750 inches in diameter shall be thoroughly cleaned in acetone and shall be placed on the inclined ramp at a distance of 8±0.1 inches from the intersection with the horizontal ramp. The ball shall be released cleanly and allowed to roll to a stop along the roving strands. The distance of roll along the horizontal ramp shall be measured to the nearest 0.1 inch. Using the same roving sample, the test shall be repeated five more times. The initial value shall be discarded and the average of the remaining five values shall be reported as the tackiness.

## 4.2.6 Bandwidth

A single sample not less than 6 feet long shall be laid flat under nominal tension of 0.5 pound per end. The bandwidth shall be measured to the nearest 0.001 inch in at least five places not less than 12 inches apart, using a vernier caliper. The average for the five values shall be reported.

#### 4.2.7 Strand Tensile Strength

Ten samples approximately 24 inches long shall be tested within 8 hours after being removed from the roll. The test shall be

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run on a testing machine using a crosshead speed of 1.0 in./min, using spool-type grips and a load weighing scale which is not more than 10 times the expected breaking strength. The grips shall be positioned so that the center-to-center distance is 6±0.25 inches. The sample shall be secured, wrapped around one spool grip at least one full turn, wrapped around the second grip at least one full turn, and the remaining end secured. The crosshead may then be adjusted to a preload of not more than 10 pounds, if desired. The crosshead shall then be operated automatically until the maximum load has been reached. The maximum load shall be recorded, and the average of the 10 values shall be reported.

- 4.2.8 Hoop Tensile Strength
- 4.2.8.1 Sample Preparation.
- 4.2.8.1.1 The samples shall be of the dimensions shown in Fig. 44.

  They shall be wound on suitably coated mandrels using a machine having the necessary tensioning, traversing, and speed controls.
- 4.2.8.1.2 The samples shall be formed by first winding a 0.030-inch-thick layer of roving in a circumferential pattern. During the winding the roving shall be maintained under a tension of 1±0.1 pounds per end. The traverse and circumferential speeds shall be selected so that, using a single strand of 20-end roving, each turn is placed so that there are 15±0.5 turns per inch and so that the mandrel is completely covered on each pass. The roving and the mandrel shall be at a temperature which is not higher than room temperature during the winding.

4.2.8.1.3 One layer of longitudinal reinforcement shall be placed over the circumferential winding. The longitudinal reinforcement shall be fabric, as described below, which has been impregnated with the resin system under test and which has been treated and stored in the same manner as the material under test. The fabric shall be wrapped over the first layer of roving, using exactly one ply. The fabric shall be placed on the mandrel so that the warp fibers are parallel to the mandrel axis. The fabric ends shall be square and shall be brought together in a butt joint. (The construction of the fabric used for longitudinal reinforcement shall be essentially as follows:

Thread count, warp, 42 per inch	150-4/2
Thread count, fill, 30 per inch	450-1/3
Thickness, nominal, inches	0.013
Weight, oz. per sq yd	15.18
Dry breaking strength, warp,	
pound/inch width	1200
Dry breaking strength, fill,	
pound/inch width	65

One fabric meeting these requirements is Style 158430, United Merchants Industrial Fabrics, New York, New York.)

4.2.8.1.4 A second layer of roving shall then be wound on the mandrel to a thickness of 0.030 inch, using the same tension, machine settings, and procedures used in applying the first layer of roving.

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- 4.2.8.1.5 The additional thickness at each end of the cylinder, as

  Fig.44 shows, shall be provided by additional circumferential

  windings applied to the basic structure, using essentially the
  same procedures used in the prior winding.
- 4.2.8.1.6 The sample shall be cured in a curculating-air oven, using the following cure cycle:

1 ± 0.1 hours at 250 ± 10 F followed by 6 ± 0.1 hours at 350 ± 10 F

The temperature shall be increased from 250 to 350 F in not more than 0.5 hour. The oven temperature shall be automatically recorded. Following the cure cycle, the cylinder shall be removed from the mandrel. The central 3-inch portion of the sample shall not be machined or finished in any way, and extreme care shall be exercised to avoid damage to the fibers.

#### 4.2.8.2 Test Procedure

- 4.2.8.2.1 Three cylinders shall be tested at room temperature by hydrostatic burst, using a test fixture essentially in accordance with that shown in Fig. 3. The fixture shall be designed so as to preclude the application of endwise loads or stress concentrations to the sample.
- 4.2.8.2.2 Prior to testing, the minimum wall thickness of the sample shall be determined, using micrometers having spherically shaped anvils. At least four equally spaced measurements shall be

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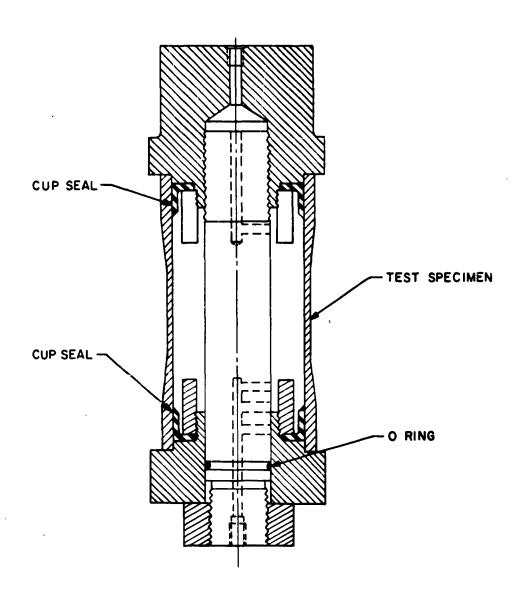


Figure 48. Hydrostatic Pressure Test Fixture

made. The thickness measurements shall be made to the nearest 0.001 inch and shall include any surface resin. All thickness shall be recorded.

4.2.8.2.3 A nonpermeable, flexible, low-strength sleeve of the proper size (latex rubber is suitable) shall be placed in the specimen which shall then be placed on the fixture and filled with water at room temperature.

CAUTION: Extreme care must be taken to ensure that all air is expelled from the cylinder before pressurizing.

- 4.2.8.2.4 The hydrostatic pressure shall be applied and increased at a steady rate of 300 psi/sec, without shock loading, until failure occurs. The hydrostatic pressure at failure shall be measured and recorded.
- 4.2.8.2.5 The thickness of the layer of longitudinal fibers shall be measured on the exposed material in the broken specimen.

  Measurements shall be made to the nearest 0.001 inch.
- 4.2.8.2.6 The hoop tensile strength shall be calculated as follows:

$$s_t = \frac{Pd}{2t_n}$$

Where  $S_{\pm}$  = Unidirectional tensile stress at burst, psi

P = Hydrostatic pressure at burst, psi

d = Mean diameter of specimen, in. =  $3 + t_{t}$ 

 $t_{\perp}$  = Minimum total wall thickness, inches

 $\mathbf{t_f}$  = Fabric thickness, inches

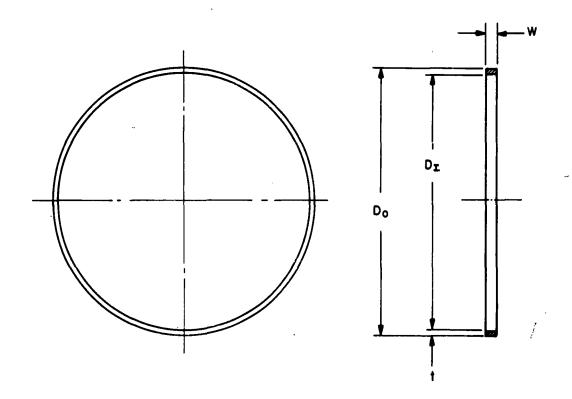
 $t_n$  = Net wall thickness, inches =  $t_t$  -  $t_f$ 

All values shall be reported. However, retests shall be made for any specimens which break at some obvious, fortuitous flaw, or for any specimens which fail outside of the central, uniform-cross-section portion of the specimen. The report shall include the average value for all valid tests.

#### 4.2.9 <u>Interlaminar Shear Strength</u>

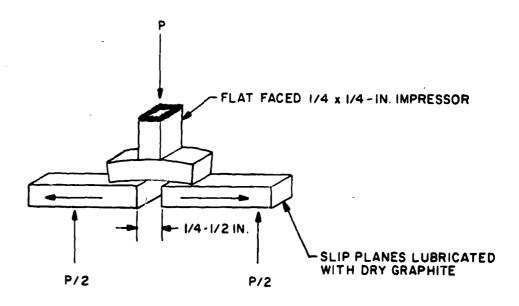
- 4.2.9.1 Sample Preparation. The samples shall be cut from a wound ring as shown in Fig. 48 and shall be of the dimensions shown in Fig. 49. The rings shall be wound on a suitably coated mandrel using the conditions specified in paragraph 4.2.8.1.2 except that the thickness of the ring shall be .250 ± .010 inches. The ring shall be cured in accordance with the cycle specified in paragraph 4.2.8.1.6.
- 4.2.9.2 <u>Test Procedure</u>. The room-temperature, interlaminar shear strength test shall be as follows:
- 4.2.9.2.1 Five samples shall be tested in a test setup which shall be essentially that shown in Fig. 50. The specimen shall be aligned so that its midpoint is centered under the loading nose and its long axis is perpendicular to the axis of the loading nose.
- 4.2.9.2.2 Before testing the thickness and width of each specimen shall be measured to the nearest 0.001 inch.
- 4.2.9.2.3 The specimens shall be loaded to failure in a calibrated testing machine at a crosshead speed of 0.05 in./min. Failure is characterized by an audible snap accompanied by a sharp drop in load.

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 $D_0 = 6.000 \pm 0.002$   $D_0 = 5.750 \pm 0.002$   $t = 0.250 \pm 0.003$   $W = 0.250 \pm 0.008$ 

Figure 49. Ring Specimen



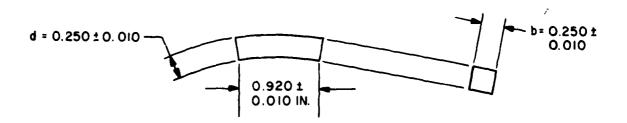


Figure 50. Horizontal Shear Specimens and Test Setup

4.2.9.2.4 The interlaminar shear strength shall be calculated as follows:

$$S_{is} = \frac{0.75 P}{bt}$$

here

S = interlaminar shear stress at failure, psi

P = breaking load, pounds

b = width of specimen, inch

t = thickness of specimen, inch

All values shall be reported. Retests shall be made for any specimens which break at some obvious, fortuitous flaw or which fail in a manner other than shear. The report shall include the average value for all valid tests.

- 4.2.9.3 The 300 F interlaminar shear strength test shall be as follows:
- 4.2.9.3.1 Five samples shall be tested in accordance with paragraph 4.2.9.2 except the entire fixture shall be enclosed in a test chamber maintained at 300 ± 5 F. The samples shall be stabilized at the test chamber temperature prior to testing. Because of the low strength of the samples, and because of the muffling effect of the test chamber, these samples may not fail with an audible noise. The loading chart of the test machine should be observed during the test. Failure is characterized by a "peak" in the load-time curve. A "shoulder" in the curve followed by further increase in load does not indicate failure. A pronounced increase in load following a peak indicates that the sample is undergoing compression.

- 4.2.9.4 The interlaminar shear strength test after water boil shall be as follows:
- 4.2.9.4.1 Five samples shall be boiled in distilled water for  $2.0 \pm 0.1$  hours. The samples shall then be transferred to room temperature distilled water and stored in water until tested. The samples shall be tested in accordance with paragraph 4.2.9.2.

#### 4.3 REINSPECTION

Twelve weeks after the lot acceptance tests, each lot of material shall be retested for resin flow and for strand tensile strength in accordance with the methods specified in paragraphs 4.2.4 and 4.2.7 of this specification. Materials which meets the requirements of paragraph 3.2 shall be maintained in storage. This procedure shall be repeated at 6-week intervals. Material which fails to meet the requirements of paragraph 3.2 on the initial or subsequent retests shall be clearly identified as unsatisfactory and shall not be used on deliverable items.

## 5. PACKAGING AND MARKING

#### 5.1 PACKAGING

The roving shall be wound with a uniform tension on a cylindrical, heavy duty winding tube  $10.9 \pm 0.25$  inches long, with an inside diameter of 3 inches. The roving shall traverse  $10.0 \pm 0.25$  inches of the tube length and shall be centered on the tube. The roving shall be wound on the tube in a regular

pattern which will permit rapid unwinding of the package on automatic machinery without whipping, fraying or other damage to the roving.

The outside end of the roving shall be secured to the package with a strip of masking tape. A rigid pad shall be placed over each end of the winding tube and pressfit tightly against the winding tube. These pads shall have a minimum thickness of 1/8 inch and the diameter shall be at least 1/4 inch greater than the outside diameter of the roll. Each roll shall be placed in a polyethylene film having a thickness of 0.003 inch (commercial). Each roll shall then be sealed in a flexible, moistureproof, sealable bag. Unless otherwise specified in the purchase order or contract, the nominal net weight of the spool shall be 15 pounds, and the minimum net weight of any one spool shall be 10 pounds.

5.1.1 The rolls shall be placed in a clean, dry container, as specified on the purchase order. The packing container shall be so constructed as to ensure acceptance by common or other carrier for safe transportation, at the lowest rate, to the place of delivery specified by the purchase order of contract.

#### 5.1.2 Shipping and Storage Environment

The material covered by this specification shall be shipped and stored at temperatures not exceeding 0 F, except it may be subjected to temporary storage at 40 F maximum for a period not to exceed 48 hours. Records shall be kept on the individual containers, showing the number of hours any roll has been in temporary storage or out of the specified storage environment.

## 5.1.3 Delivery Date

0

**j** :

Material which has been impregnated more than 30 days prior to the delivery date shall be subject to rejection without recourse to, and regardless of the results of, any lot acceptance tests.

#### 5.2 MARKING

Marking shall include but not be limited to the following:

## 5.2.1 Winding Tube Identification

Each winding tube shall be clearly and permanently marked as follows:

- 1. Number of this specification, including type and class designation
- 2. Manufacturer's name and designation
- 3. Lot number of impregnated roving
- 4. Spool number
- 5. Date of impregnation

## 5.2.2 Package Marking

Individual roll containers shall be clearly and permanently marked with the following information:

 Number of this specification including type and class designation.

- 2. Manufacturer's name and designation
- 3. Lot number of impregnated roving
- 4. Spool number
- 5. Date of impregnation
- 6. Net weight
- 7. Rocketdyne Purchase Order Number

## 5.2.3 Warning Markings

Each roll container and the exterior packing shall be prominently and permanently marked with a storage warning essentially worded as follows:

- 1. Temperature sensitive materials
- 2. Keep away from heat
- 3. Store at 0 F maximum